TOPICS OF THE MONTH

Divide and conquer?

MILITARY strategists often claim that their maxim 'divide and conquer' is useful not only in overcoming an opponent in the field of battle but also in every other sphere. The essential truth of this saying may accurately reflect the state of contemporary human affairs, but the very opposite is surely happening in science and technology. There the trend is to unify rather than divide because only unification can demonstrate certain similarities between various systems. A good example of increasing comprehension as a result of unification can be seen in the field of mass-transfer operations. Hitherto unit operations in chemical engineering were treated as self-contained entities; distillation, evaporation, and absorption, for example, all grew up in different circumstances and previous analyses of these operations were often quite pragmatic. Lately a tendency has grown to group those operations involving similar phases into one category.

Taking the solid-fluid phase of mass transfer, operations in this category include adsorption, drying and leaching. Whereas adsorption involves contact of solids with either liquids or gases with mass transfer in the direction fluid to solid, drying involves gas-solid and leaching liquid-solid contact with mass transfer in each case in the direction solid to fluid. Thus, theoretically at least, similar plant used for gas-solid or liquid-solid contact in adsorption should be correspondingly useful for drying and leaching. In practice, however, different types of equipment are to be found in all three operations—this undoubtedly is due to the fact that each of them has had a different historical background. Drying, for example, has been used for centuries in the manufacture of pottery and ceramics—drying ovens were designed simply to reduce moisture in the clay which had already been pre-formed. Similarly, leaching, although not as ancient an art as drying, has been used for a long time in extraction of fats from groundnuts and in many other processes in food manufacture. In contrast to this, adsorption is a relative newcomer and only during the past 30 years has the importance of solvent recovery based on adsorption been fully understood, with the result that unification of the three operations is becoming increasingly fruitful.

This is clearly demonstrated in Dr. Molyneux's article in this issue, on the design of a continuous counter-current adsorber, using well-known principles previously applied in leaching and gas absorption. It is amazing that similar work has not been reported to date; but it is one more proof that water-tight compartments are slowly being merged.

The industrial importance of solvent recovery cannot be over-stressed. We hear much nowadays about solvents recovered being equivalent to money saved, but this has certainly not been fully appreciated by those who could best benefit by it. The total consumption of solvents by British industry in 1959 was approximately 700 million gal. One of the largest applications of solvent recovery is in the production of regenerated cellulose and it is claimed that the total amount of solvents recovered in this industry during last year was only 9 million gal. Although comparison between these two figures would be completely inaccurate without further breakdown, it does seem that there is at present an enormous gap between the potential and actual applications of solvent recovery. Here, of course, is the great challenge to adsorption within the realm of mass transfer. Only by further extensions of chemical engineering principles already successfully applied to distillation, evaporation and leaching will industry efficiently and effectively benefit from solvent recovery.

Blue water-gas

THE possibility of eventual shortage of good-quality coke used to manufacture blue water-gas has once again created interest in the complete gasification of coal. Already 10 years ago the Gas Council initiated work on new methods for producing blue water-gas. At that time a comprehensive survey of all known gasification methods was carried out and, as a result of this survey, attention was focused on a process known as the slag-bath generator, developed in Germany. This is to be intensively studied in a new pilot plant to be built by the North Thames Gas Board at Bromley.

The process involves forcing powdered brown coal together with air, oxygen or steam into a bed of molten slag which produces blue water-gas continuously. Modifications have had to be made in this country due to the absence of large quantities of brown coal; thus the pilot plant will use powdered low-rank black coal as a fuel, and air instead of oxygen. Because of this, the slag-bath generator will consist of a twin-shaft system and a divided slag bath, in one part of which coal is burned in air with combustion products escaping up one of the shafts. In the other part, coal will react with steam in the presence of molten slag, heated by combustion in the first half of the bath, to produce blue water-gas. It is intended to keep the slag circulating under a dividing curtain so that heat generated on the combustion side is carried into the gas-making side to provide the heat required in the process. This is a complete gasification process and the only by-product formed is slag. The pilot plant, which will cost about £225,000, is expected to produce 2 million cu.ft./day of blue water-gas.

The intention of the Gas Council is that this cheap blue water-gas will be mixed with rich gas—such as methane from oil gasification or tail gases from refineries—and so provide town gas.

Manufacture of fatty acids

GLYCERIDE oils and fats continue to be the major source of fatty acids, and fat splitting—hydrolysis of glycerides to give glycerol and fatty acids—is still an important process employed in this reaction. Precise knowledge about physico-chemical properties of fatty acids and the mechanism of the fat-splitting reaction have resulted in a vast improvement in these processes. For example, the batch autoclave catalytic method of fat splitting has been developed into a continuous high-pressure method and the expensive operation of separating fatty acids by hydraulic pressing has been replaced by low-temperature solvent crystallisation as represented by the *Emersol* process. At a recent symposium on developments in the manufacture and utilisation of fatty acids held in the Department of Chemical Technology, University of Bombay, S. N. Modak surveyed recent advances in fat-splitting technology. The most important processes used are *Twitchell*, low-pressure autoclave and high-pressure autoclave, both batch and continuous.

The *Twitchell* process, which is still widely used, is in constant need of active catalysts. Originally, *Twitchell* catalysts were prepared by sulphonating a mixture of oleic acid and aromatic hydrocarbons. These were gradually replaced by the sulphonated products obtained as by-products in petroleum refining. Catalysts of the alkyl-aryl type are the most efficient and are reputed to give a 90% split in about 8 hr. The splitting process became more efficient through the use of autoclaves by means of which higher reaction temperatures are possible. The present trend in fat splitting is to use higher temperatures of the order of 220 to 260°C., corresponding to steam pressures of 350 to 700 p.s.i. This has been made possible because of newly developed molybdenum-containing stainless steels which can withstand the corrosive action of fatty acids at high temperatures. At such temperatures, without the use of catalyst, 90% split can be obtained in 2½ hr., compared with 10 hr. in low-pressure autoclave and 30 to 40 hr. in the *Twitchell* process.

The crude fatty acids obtained after splitting contain a variety of fatty acids as well as unsplit fat, unsaponified matter, colouring matter and impurities present in the original oil. The following processes are used to refine the crude fatty acids:

(*a*) Simple distillation.
(*b*) Fractional distillation.
(*c*) Separation of unsaturated from saturated fatty acids.

In view of the fact that fatty acids are heat-sensitive it is most important to avoid heating in excess of 15 min. during fractionation. The oldest and still most widely used process of separating stearic and oleic acid is by pressing a suitable blend of tallow fatty acids. When the blend contains stearic and palmitic acids in the ratio 55 : 45, the cake to be pressed becomes quite porous and stearic acid can be squeezed out at lower pressures. This is fast being superseded by the *Emersol* process.

Buckling in the RB reactor

INTERESTING determinations to measure buckling in a heavy water natural uranium reactor were recently carried out by N. M. Raisic and others at the Boris Kidrich Institute of Nuclear Sciences, Yugoslavia. The studied lattice core was square with a 12-cm. pitch. The fuel elements, cylindrical uranium rods having a diameter of 25 mm., were canned in 1-mm. thick aluminium.

In their experiment, buckling (which is the curvature needed for a reactor to be critical) was determined on the basis of thermal neutron flux distribution in the core measured by means of indium and dysprosium foils.

Originally the heavy water critical assembly RB of the Institute was designed bare, to allow for an exact determination of buckling by measuring the geometrical characteristics of the system and only introducing small corrections due to the presence of the tank walls, the lid and the supporting platform which act as neutron reflectors. The results stress, however, that the assembly cannot be taken as bare because construction elements influence the boundary conditions and act as reflectors. Their influence is rather small in this case and does not affect the total height of the heavy water by more than 10 mm. The total buckling can be shown as the sum of the horizontal and vertical bucklings

$$B^2 = B_h^2 + B_r^2 = (8.516 \pm 0.02)m^{-2}$$

where B_r and B_h are the bucklings in the vertical and horizontal direction respectively.

There are some published data available on the buckling of heavy water uranium assemblies and it is well known that, except for the Canadian and French, measurements do not agree well with one another. The American value for the buckling of the lattice with 1-in. diam. uranium rods and a pitch of 4.5 in. is 8.47 m^{-2}. For a lattice of 2.54-cm. diam. rods and 12-cm. pitch the Swedish exponential experiment gave 8.66 m^{-2}. The Yugoslav value for the lattice of 2.5-cm. rods and 12-cm. lattice spacings is practically the same as the American but agrees less well with the others.

East European chemical industry

ACCORDING to the United Nations' report 'Economic Survey of Europe, 1959', the Soviet Union plans to invest about $25 billion during the next seven-year plan, 1959-65. The Russians are aiming at a chemical production index of 200 during the plan period, taking the 1958 index as 100. A significant trend is shown by the planned increase in capacity for synthetic materials rather than heavy inorganic chemicals which the Soviet Union has largely produced hitherto. Fibres and plastics, for example, will absorb 39% of the total investment in that period, compared with 20% during 1952-58.

In Eastern Europe, East Germany will be placed immediately behind the Soviet Union as a producer of fibres and plastics. Much of the increase of East Germany's chemical production is to be directed

towards export markets and it is expected that total chemical exports will double during 1958-65. An enormous expansion is predicted for Czechoslovakia where the contrast between the rate of growth in the chemical and other priority industries is more noticeable than in previous years. Increases are also forecast in Hungary where emphasis is being placed on chemical fertiliser production, and completion of new projects is expected to result in rapid growth of output in mineral oils, natural gas and cement. Poland is the only country in Eastern Europe with identically planned rates of growth for both consumer and investment goods up to 1965. It is expected that expansion will be particularly marked for brown coal and natural gas, sulphuric acid, synthetic fibres, plastics and synthetic rubber.

This total planned increase in productivity has been made possible only by the rising imports of oil and natural gas as well as exploitation of available domestic supplies of these fuels. Although a certain amount of specialisation for each country is envisaged, the overall aim is still to build a substantial and highly diversified chemical industry which in 1965 is expected to be three times as large as in 1958. One cannot really begin to prophesy how this state of affairs will affect both the export market and internal economy of Western Europe.

Burst-strength predictions

IN the April issue of CHEMICAL & PROCESS ENGINEERING J. A. Rhys pointed to the lack of available data on long-term ageing properties of plastic materials. This is not surprising, because some of these materials have only been in production for a few years. As a result, many manufacturers have had to extrapolate data in order to predict ageing properties of up to 20 years—obviously an unreliable procedure.

An elegant method of calculating long-term ageing properties in plastics has been described by S. Goldfein in *Modern Plastics* (**37** (8), 127). The author has developed a parameter K which may be used to predict creep and rupture properties:

$$K = \frac{T_o\, T}{T_o - T}\ (20 + \log\ t)$$

where T_o = zero strength temperature
T = operating temperature
t = time.

Experimental data are given to support this parameter, which in the case of glass-reinforced plastics approximates to

$$K = T(20 + \log t)$$

which is the Larson and Miller parameter.

It is claimed that, regardless of the types of stresses set up, bi-axial, hoop or otherwise, failure is always by separation of the molecules of the material, so that in the case of complex stresses results may be predicted from graphs. Thus for pipes a curve of burst strengths *versus* K drawn from short-time tests at elevated temperatures would enable long-term properties to be calculated.

Increasing pipelines

THE use of pipelines for transporting crude oil is so extensive that there are now more than a quarter of a million miles of oil-carrying pipelines stretching across the western world. Development has been mainly in the U.S.A., where this system was originally developed by the oil industry, and until comparatively recently this method of transport was not widely utilised in Europe, where crude oil and refined products alike have generally been delivered by tanker and conveyed to inland destinations by road, rail and barge. Now, however, plans are afoot for several projects which, within a few years, will transform the methods of transporting crude oil in Western Europe.

Four new refineries will be constructed in the vicinity of Strassbourg in France and Karlsruhe in Western Germany. To provide the necessary crude oil, pipelines are being laid which will handle about 35 million tons annually in the initial stages. The first of these lines—from Wilhelmshaven to Wesseling, near Cologne—came into operation last year. Another, from Rotterdam to Venlo, Wesseling and Wesel, is scheduled to commence operations this summer. In the beginning each will handle about 9 million tons of oil, rising subsequently to 22 and 20 million tons respectively.

An even more ambitious project is the trunk line being laid from Lavera, near Marseilles. This will cross through France to Strassbourg and across the border of Germany to Karlsruhe—traversing a distance of nearly 500 miles. It is due for completion before the end of 1962, when it will have a capacity of 10 million tons, with a maximum of 30 millions being provided for in the future. There are other lines scheduled, one from Genoa through Northern Italy to Aigle, and another one from Aigle (in Switzerland) to Munich.

The wide variation between the initial throughput of the various pipelines and their ultimate capacity is an indication of the extent to which refining capacity may ultimately be expanded. At present inland refineries in Western Europe can process about 45 million tons of crude oil a year altogether, although if coastal plants are included the five countries concerned (France, Western Germany, Netherlands, Italy and Switzerland) treated more than 100 million tons last year, as compared with their domestic consumption of about 70 million tons. While it is likely to be many years before these pipelines will be used to their maximum capacity, it is best to make ample provision for expansion now.

The new arteries that are even now being created are likely to have far-reaching repercussions that will extend beyond those European countries most obviously affected. The advantage of these pipelines is that they are the most economical means of conveying the very large quantities of crude oil required, and the fact that existing methods of transport are already being strained to their limits and could not easily be adapted to handle the substantially larger volume of oil likely to be needed in the years to come.

Thus, introduction of trunk pipelines will inevitably affect existing transport methods—whether by road, rail or canal.

Sodium heat exchanger

A UNIQUE feature of the three heat exchangers recently installed at the *Enrico Fermi* atomic power plant on Lake Erie, Michigan, is the sine curve in the tubes. This compound curve is necessary because of space limitations and flow direction in the units accommodates differences in thermal expansion in the tubing. Furthermore, the tube bundles can be easily removed from their shells, thus facilitating maintenance work. In addition to being removable the bundles have a gasketed joint which separates the primary from secondary sodium fluids. Ten-inch-long springs act as load members for setting and sealing this joint. The heat exchangers contain more than 19 miles of type 304 stainless-steel tubing. The shells are 31 ft. long and 4 ft. 4 in. in diameter, including 16 in. of shielding. One completely assembled unit will weigh 130,000 lb.—this makes them the largest heat exchangers ever constructed.

Heat is removed from the *Fermi* reactor by liquid sodium which circulates through the core and blankets to the heat exchangers. A second sodium system transfers the heat from the exchangers to steam generators for the production of steam to operate a turbine generator.

Each of the three exchangers is designed to transfer about 489 million B.Th.U./hr. at flow rates of 12,500 gal./min. In the exchanger, primary sodium coolant flows by gravity from the reactor at a temperature of 900°F. through the shell side of the unit to the pump, from which it is returned to the reactor at 600°F. Secondary sodium from the steam generator is pumped at a temperature of 520°F. through the downcomer in the exchanger and through 1,860 bowed tubes where it is heated to 820°F. by the primary sodium and is then returned to the steam generators. Three primary coolant loops and three secondary coolant loops transfer the heat from the reactor.

Plant constructors' anxiety

THE annual report of the British Chemical Plant Manufacturers Association for 1959, which was recently published, reflects some of the unease felt by the chemical plant construction industry last year. Imports of chemical and gas machinery plant in 1959 came to 904 tons at a value of £0.84 million. This is considerably less than for 1958, which totalled 1,296 tons at a value of £1.28 million. In contrast to this, exports rose slightly during the same period, as can be seen from the following figures:

	Quantity in tons		*Value in £ million*	
	1958	1959	1958	1959
Gas and chemical machinery	12,219	7,424	5.26	5.45
Plastics and rubber working machinery	7,832	14,714	4.68	9.76
Total chemical and allied plant	28,527	30,830	14.70	20.17

This overall increase, however, is almost entirely due to the very steep rise in exports of plastics and rubber working machinery which had a large boost in 1959 due to the Russian tyre plant which a British consortium is currently erecting.

The economic division of Europe is beginning to cause anxiety to chemical plant constructors. The report pointed out that, during the preliminary stages of the discussions leading to the European Free Trade Association (the outer seven), the government sought the views of the B.C.P.M.A., which indicated that the chemical plant industry was prepared to acquiesce in these negotiations on the understanding that the formation of some acceptable trading association with the European Economic Community would thereby be facilitated. This qualification was important to many members who considered that E.F.T.A. offered no immediate direct benefit on its own. With the exception of Austria and Norway, other countries in the outer seven have much lower tariffs than the United Kingdom and would therefore benefit much more from a percentage reduction in tariffs. That the volume of trade in chemical plant between the U.K. and other members of the outer seven is not high has been shown by the exports to these countries which in 1958 totalled £0.45 million or 3% of total exports. During the same period 8% was exported to the six countries of the European Economic Community.

These figures speak for themselves and it would seem at present that the only hope is to develop the outer seven export market which has a larger potential than would seem from the figures quoted above. Nevertheless, one does hope, for the benefit of the industry at large, that the government has not and does not intend to burn all its bridges with the E.E.C.

Science for the masses

WHO would have believed that the periodic table, this chemical *sanctum sanctorum*, could ever be performed as a song? We scarcely trusted our ears when a well-wishing friend played us the latest recording of Harvard mathematician-turned-entertainer, Tom Lehrer, on the periodic table. The song was prefaced with a thoughtful comment that to most people chemical periodicity *per se* was far too dry and badly needed livening up. As a result 102 elements were declaimed, to suitable pianoforte accompaniment, at a tempo of *allegro molto vivace*. Mr. Lehrer's grouping was solely according to rhythmic merit—quite pleasing to our sense of jingle, though we doubt whether altogether acceptable to the International Union of Pure and Applied Chemistry. This homage to chemistry concluded with a most appropriate summary, and here we quote from memory:

> 'These are all the elements known to Harvard.
> 'There may be others but they haven't been discovered.'

As a first step in bringing science to the masses this seems to us quite unsurpassed. We eagerly await further improvisations in the arena of the classroom. Then grubby fifteen-year-olds will cease to bewail the difficulty of memorising the relationship between beryllium and francium, but, on the contrary, will tap it out to the latest rock rhythm.

Adsorption and its Application to Solvent Recovery

By D. J. Spikins,* Ph.D., B.Sc., A.M.Inst.F.

The difference in mechanism between absorption and adsorption is defined and the two types of adsorption are discussed. Work on adsorption rates and equilibria is mentioned and a method for the calculation of heats of adsorption is given. A review is given of theories of adsorption, and the general principles of the application of adsorption to solvent recovery are described.

ADSORPTION is a unit operation in which materials are concentrated on the surfaces of solids, making use of surface forces. There is a clear distinction between adsorption and absorption. In the case of adsorption there is commonly no chemical reaction between the solid adsorbent and the adsorbate, or material being adsorbed. In the case of absorption there is a phase change or chemical reaction which is of a more permanent nature.

When gasoline is removed from natural gas by contacting the natural gas with activated carbon, the gasoline is concentrated on the surface of the carbon. The gasoline is held by surface forces and may be easily removed by raising the bed temperature and distilling off the gasoline.

In the absorption process for gasoline removal the natural gas is brought into contact with an absorption oil, which is a fraction between heavy gasoline and light gas oil, in a column. There is then solution of the gasoline in the absorbent, and any separation must be by fractional distillation.

These examples show that, in practice, there is no difficulty in distinguishing between adsorption and absorption, although the distinction is less clear when chemisorption is involved.

Desorption, the reverse of adsorption, is a mass-transfer operation in which material is transferred from a solid to a gas or a liquid, and is therefore similar to drying and leaching. While it would be possible, in theory, to use the same apparatus for adsorption as for leaching and drying, the processes have developed independently. It is possible that useful ideas could be obtained from similar operations in order to solve particular problems, and also study of the operations is simplified by recognising similarities between them.

An adsorbent may be considered to be a solid foam, in which the external surfaces are very much smaller in area than the internal surfaces. The surface area of a gas-mask charcoal has been estimated to be 1,000 sq.m./c.c.[1] The internal area is made up of microscopic pores and the adsorbate diffuses into these. Provided that the adsorbate does not enter the field of force between the atoms or molecules of the solid, it is said to be adsorbed. If the adsorbate does penetrate this field of force, it is considered to have been absorbed.[2]

Desiccants such as sulphuric acid, calcium chloride and phosphorus pentoxide remove moisture by the formation of hydrates and a phase change occurs following the formation of the highest stable hydrate, resulting in a change in the physical and chemical properties of the absorbent. On the other hand, a solid adsorbent will remain in the same state, having the same physical and chemical properties, until it is saturated with moisture.

Types of adsorption

When the atoms or molecules of a gas or vapour are adsorbed on the surface of a solid adsorbent, they are held on the surface in two different ways. If there is a weak interaction between the gas or vapour and the adsorbent, the process is called physical or van der Waals adsorption. In this case, the forces are those forces which produce condensation in liquids, known as van der Waals forces. In the second case a much stronger interaction occurs and the process is called chemisorption or activated adsorption, because the interaction is similar to chemical reaction and requires activation energies. It is of great importance in catalysis. Desorption is much easier following physical adsorption than chemisorption.

The atoms and molecules in a solid adsorbent, as in any other solid, are held together by various forces in such a way that the forces are unbalanced and occur in a manner that makes the inward force greater than the outward force. A solid will therefore exhibit surface tension, which is reduced by the forces of gas and vapour molecules striking the surface and balancing the forces of the surface atoms, which previously were tending to reduce the surface area. The surface energy, which is a function of surface tension and surface area, will be decreased and hence adsorption is a spontaneous process in that it reduces the surface energy of the adsorbent.

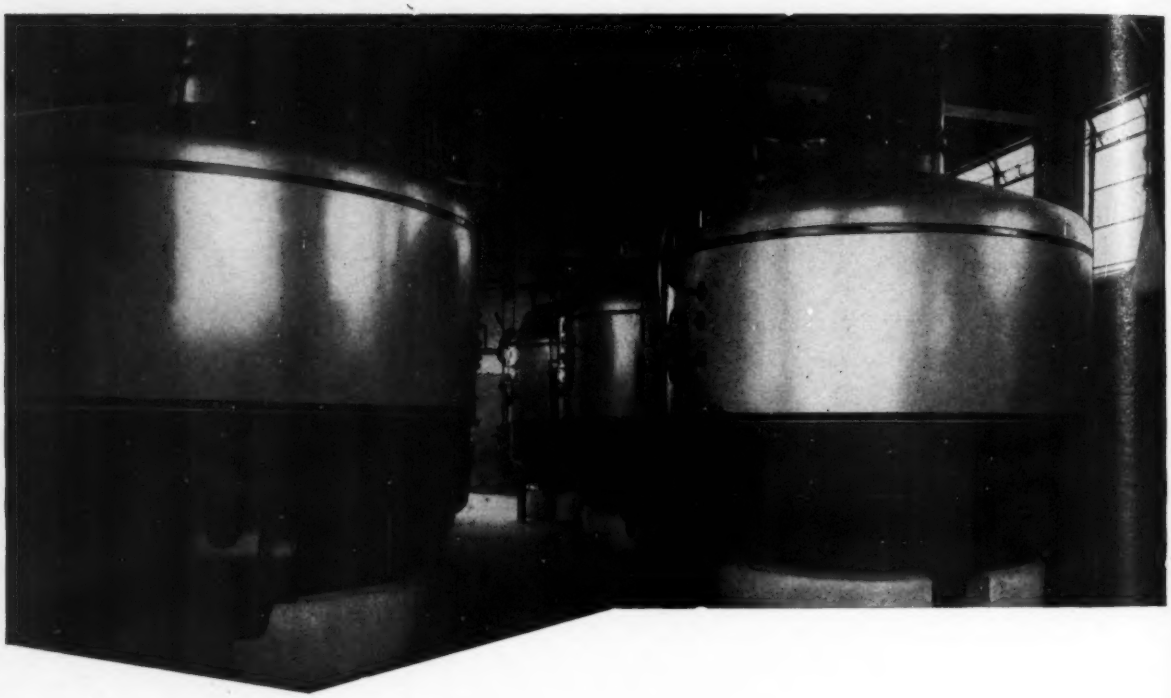
The heat of adsorption may be used to determine whether physical adsorption or chemisorption is taking place. The process is an exothermic process, which follows from experimental work that shows that adsorption decreases as the temperature rises. The heat of adsorption for physical adsorption is of the same order as the heats of liquefaction of the gases considered, while in chemisorption the heat of adsorption is similar to the heats of chemical reaction.

Mantell gives the heat of physical adsorption of hydrogen on zinc oxide at 0°C. as 1,900 cal./mole and the heat of adsorption for chemisorption at 300 to 444°C. as 20,000 cal./mole.

* *Department of Chemical Engineering, College of Technology, Loughborough.*

CARBO-UNION-WHESSOE

Recovery Plant



Whessoe Ltd. design and manufacture Carbo-Union-Whessoe Activated Carbon Recovery Plant for the purification of gases and the recovery of vapour phase solvents in accordance with the agreement between B.C.N.U. Ltd. and Whessoe.

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The results were obtained originally by Taylor and Sickman.[3]

In the case of physical adsorption the hydrogen would be recoverable by evacuation, while at the higher temperatures the hydrogen would not be recoverable, being held by chemical bonding.

Since the heats of physical adsorption of gases on charcoal are greater than the heats of condensation of the gases it may be assumed that the forces between the adsorbent and gas molecules are greater than the forces between the molecules of the gas when it is liquefied.

The difference between physical and chemisorption is illustrated by Glasstone, Laidler and Eyring[4] using a potential energy diagram (Fig. 1). A gas is considered which may be adsorbed by either type of adsorption. A gas molecule is brought towards the surface of the adsorbent and at the distance corresponding to the equilibrium value there is a van der Waals attraction, while if the distance is decreased further the repulsive forces will increase. The equilibrium value is shown on curve I where the heat of adsorption is ΔH_W. In the case of chemisorption the equilibrium state is represented by the minimum value of curve II where the heat of adsorption is ΔH_A. At the point A, the two curves cross, but there will be a gradual change, as shown, due to resonance effects. The height E represents the activation energy for activated adsorption. Some dissociation of the adsorbed gas molecules is considered to take place and this is shown in Fig. 1 where, at the right-hand end of the curves, curve II is higher than curve I.

Adsorption rates and equilibria

Most experimental work on adsorption has been concerned with equilibria, largely to obtain evidence in support of the theories that have been produced and because adsorption rates are frequently very high. On the other hand, it is the adsorptive capacity and rate of adsorption which is important in practice. Because adsorption is a rapid process, a solvent recovery plant can completely remove solvent vapour from an air stream without the limitations imposed by equilibrium considerations in operations such as absorption and condensation. The adsorbent is usually present in excess and is not allowed to reach equilibrium at the outlet end of the adsorber, because this would mean that some solvent would escape in the effluent gas.

Lamb, Wilson and Chaney[1] measured the rate of adsorption of chlorpicrin on a charcoal for a military gas-mask and showed that 99.9% of an initial concentration of 7,000 p.p.m. of gas was adsorbed in 0.03 sec.

The experiments of Bergter[5] are also described by Mantell.[2] In these experiments it was shown that, while the initial rate of adsorption was very high, equilibrium was sometimes only reached after many hours. The slower process has been ascribed to chemisorption, chemical reaction and the inability of molecules to reach the surface of the adsorbent where the pores are very long and narrow.

Taylor[6] introduced the concept of activated adsorption in order to explain both decreases in adsorption rates which were found at high temperatures and to explain the variation in amount of adsorption with changes in temperature.[7] With physical adsorption the amount decreases with temperature at low temperatures, but increases as the temperature becomes higher due to the increasing rate of chemisorption. Finally, the amount adsorbed begins to decrease as still higher temperatures are reached.

The measurement of the amount of vapour adsorbed by a known weight of adsorbent may be carried out in a number of different ways. Mantell[2] describes in detail methods suitable for different purposes. These include the classical volumetric method and the use of the sorption balance described by McBain and Bakr,[8] which is a gravimetric method which overcomes the difficulty of determining the volume unoccupied by the adsorbent in the bulb containing a known weight of adsorbent. There is also a dynamic method which is used with porous adsorbents in which the inlet and outlet gas streams are analysed at fixed intervals. In addition, Mantell describes a number of indirect methods.

Adsorption isotherms

Adsorption data may be plotted as isotherms, isobars and isosteres, in which temperatures, pressure and adsorbate concentration, respectively, are parameters. The most usual method is the adsorption isotherm since this method of plotting is the most convenient way of representing experimental data. The isotherm is represented by the equation

$$a = t(p^*) \quad \ldots\ldots\ldots\ldots (1)$$

in which a is the amount adsorbed/unit weight of adsorbent, p^* is the equilibrium pressure and the temperature, t, is constant.

Treybal[9] gives a series of adsorption isotherms for methane, benzene and acetone being adsorbed on an activated carbon (Fig. 2). The curves obtained with other adsorbents and with activated carbon of a different type would be different. For this reason, data

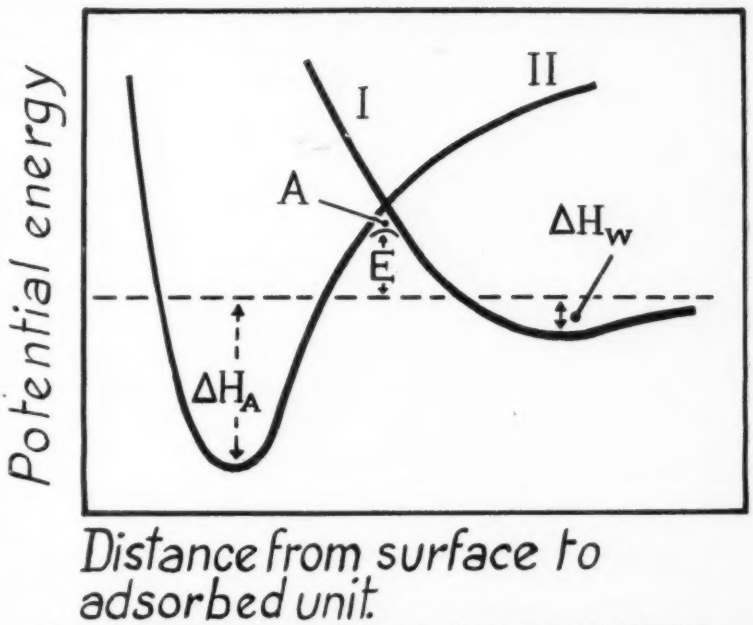

Fig. 1. Potential energy curves for van der Waals activated adsorption.

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This is the ONLY continuous solvent recovery plant of its kind

In this SUTCLIFFE, SPEAKMAN Continuous Solvent Recovery Unit, Active Carbon manufactured by SUTCLIFFE, SPEAKMAN is used to extract solvents from solvent-laden air and from other gases.

The type of SUTCLIFFE, SPEAKMAN Continuous Solvent Recovery Unit illustrated here consists of a horizontally disposed segmented adsorber which rotates slowly and continuously in a chamber into which is directed the solvent-laden air stream. The segments pass in turn through a positively segregated steaming zone, in which the isolated carbon bed releases the solvent which it has adsorbed in the adsorption chamber. It is the *only Recovery Plant on the market* which is both continuous and automatic in operation, and which has these features:—

- ✳ *The continuous cycle of adsorption and release of the solvent from the carbon.*
- ✳ *Only one adsorber necessary for this process.*
- ✳ *Even consumption of steam and water: no peak demands.*
- ✳ *No cyclic operation valves.*
- ✳ *The large amount of work done by the comparatively small amount of active carbon.*
- ✳ *The very small amount of floor space required.*
- ✳ *Simplicity of installation: this is truly a "packaged unit".*
- ✳ *Easy maintenance: all parts are readily accessible, and access doors are fitted with quick release fasteners.*

This continuous unit represents an extension of the range and types of solvent recovery plant made by SUTCLIFFE, SPEAKMAN:— their well-known Multiple Adsorber Plant continues to be popular and highly successful. But only SUTCLIFFE, SPEAKMAN can offer alternative designs of solvent recovery equipment.
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required for a particular process need to be determined for the adsorbent which is to be used. It can be seen from Fig. 2 that the amount of acetone adsorbed at any pressure decreases as the temperature rises. This result follows from the fact that adsorption is an exothermic process.

Fig. 2 also shows that, at the same temperature and equilibrium partial pressure, benzene is adsorbed to a greater extent than acetone. Structural differences in compounds will affect the adsorption isotherm of a substance, but it has been found that high molecular weight substances with low critical temperatures are most readily adsorbed. The permanent gases are only slightly adsorbed and this fact is shown by the isotherm for methane on Fig. 2.

Hysteresis effect

It would be expected that, since the isotherms are equilibrium curves, any point on these curves could be reached by either adsorption or desorption. In practice a hysteresis effect may often be observed and, in these cases, the equilibrium partial pressure for a given adsorbate concentration is always lower for desorption than for adsorption. A number of attempts to explain this phenomenon have been made. Mantell[2] summarises the investigation of Cohan[10] in which three possibilities were considered. The first suggests that hysteresis is caused by incomplete wetting of the surface due to a film of gas, but it was considered that this was not acceptable, since the hysteresis effect is only observed on part of the isotherm and persists when the gaseous impurity is removed. The second theory considered that pores with constricted necks would cause hysteresis. This was thought to contribute partially to the phenomenon, but in certain cases the structure may consist of pores that widen towards their bases and behave as open pores. In the open-pore theory it is assumed that there is a delay in meniscus formation and that this causes hysteresis effects. This theory was supported by predictions, confirmed by experiments, of the point at which the hysteresis loop begins.

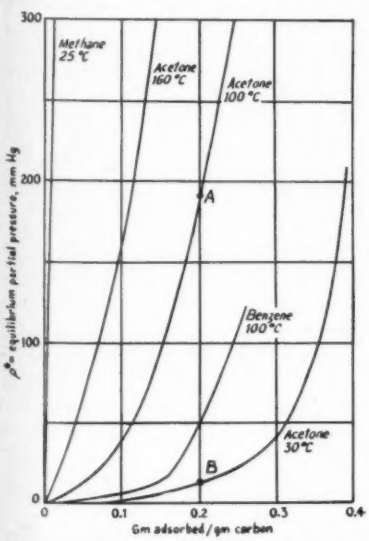

Fig. 2. Equilibrium adsorption on an activated carbon.

(Figs. 2, 3, 4 and 5 by permission from 'Mass Transfer Operations' by Treyball. Copyright 1955, McGraw-Hill Book Co. Inc.)

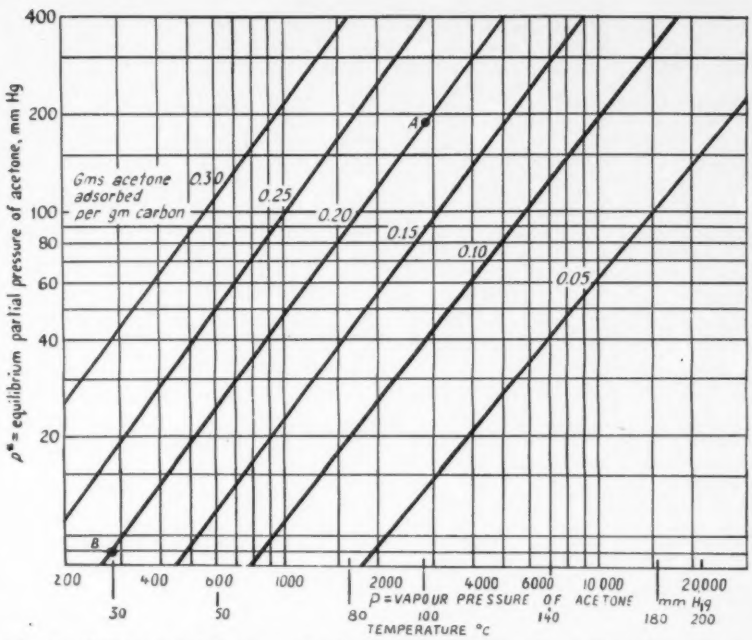

Fig. 3. Reference-substance plot of equilibrium adsorption of acetone on an activated carbon.

Heats of adsorption may be determined by the use of the reference-substance method of showing results.[11] Treybal[12] shows a graph of the adsorption of acetone vapour on activated carbon using acetone as the reference substance (Fig. 3). The vapour pressure and corresponding saturation temperature for acetone were marked on the abscissa and the equilibrium partial pressure of acetone was marked on the ordinate. The lines of constant adsorbate concentration are usually straight lines and only two points are required to establish each.

For the concentration 0.2 g. acetone/g. carbon, two points are obtained from Fig. 2. Point A corresponds to a temperature of 100°C. and an equilibrium partial pressure of 190 mm. Hg, and point B corresponds to a temperature of 30°C. and a pressure of 9 mm. The other isosteres may be obtained in the same manner. In order to obtain Fig. 3 completely from Fig. 2, data would be required at a temperature of about 80°C.

The differential heat of adsorption ($-\overline{H}$) is defined as the heat liberated when unit mass of vapour is adsorbed on an adsorbent which already has adsorbed some adsorbate, and is present in such quantity that the further adsorption does not change the adsorbate concentration.

The differential heat of adsorption is given by

$$\frac{d\ ln\ p^*}{d\ ln\ P_S} = \frac{-\overline{H}\ M}{\lambda_r\ M_r} \quad \ldots\ldots\ldots (2)$$

in which $\frac{d\ ln\ p^*}{d\ ln\ P_S}$ is the slope of the isostere, λ_r is the latent heat of the reference substance at the temperature for which $-\overline{H}$ is required and M and M_r are the molecular weights of the vapour and reference substance.

Frequently the vapour and reference substance are the same and equation (2) becomes

$$-\overline{H} = \lambda_r\left(\frac{d\ ln\ p^*}{d\ ln\ P_S}\right) \quad \ldots\ldots\ldots (3)$$

For an adsorbate concentration of 0.20 g./g. of carbon and at a temperature of 40°C., the heat of adsorption

may be found from equation (3). The slope of the isostere is 1.31 and the latent heat of acetone at 40°C. is 128 cal./g., hence $-\bar{H} = 167.5$ cal./g. of vapour adsorbed.

The integral heat of adsorption at any concentration of adsorbate is defined as the enthalpy of the adsorbate-adsorbent concentration minus the sum of the enthalpies of unit weight of the adsorbent and of such a quantity of the pure adsorbed substance to give the required concentration, all at the same temperature. It may be referred either to the vapour or the liquid.

The integral heat of adsorption, $\Delta H'$, may be found by integrating the differential heat of adsorption from zero to the required concentration, the values of the differential heat of adsorption being found from equation (3).

$\Delta H'$ is given by

$$\Delta H' = \int_0^x \bar{H}\, dx \quad \ldots\ldots\ldots (4)$$

in which x represents the adsorbate concentration. The integral heat of adsorption referred to the liquid state is given by

$$\Delta H = \Delta H' + \lambda x \quad \ldots\ldots\ldots (5)$$

in which λ is the latent heat of vaporisation.

Heats of adsorption are important in that this heat must be removed during an adsorption operation, to prevent the temperature of the bed rising and causing a decrease in the adsorptive capacity of the bed.

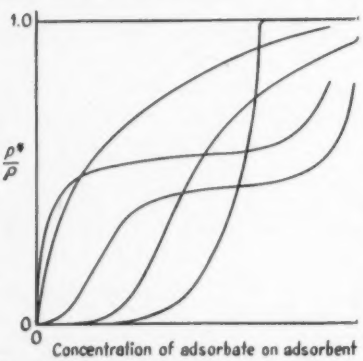

Fig. 4. Types of adsorption isotherms for vapours.

Theories of adsorption

Brunauer *et al.*[13] found five different types of physical adsorption isotherms. They are shown on Fig. 4. The limiting concentration of adsorbate on the adsorbent for a gas below its critical temperature or a vapour occurs when the equilibrium partial pressure equals the saturation vapour pressure of the vapour at the temperature of the experiment. Concentrations greater than this may only be obtained by liquefaction.

Adsorption data are concerned with the distribution of the adsorbate between the gas and solid phases and should conform to an equation of the type

$$x = K p^* \quad \ldots\ldots\ldots\ldots (6)$$

This equation may be considered as a limiting value for adsorption at very low concentrations. The Freundlich exponential equation was introduced to cover data outside this range. It is usually expressed as

$$x = K p^{*n} \quad \ldots\ldots\ldots\ldots (7)$$

in which x is the concentration of adsorbate/unit weight of adsorbent, p^* is the equilibrium partial pressure and K and n are experimentally determined constants, n being a fraction.

Equation (7) may be tested by writing it in a straight line form

$$\log x = \log K + n \log p^* \quad \ldots\ldots (8)$$

when it is found to satisfy the data over a very limited range in most cases. However, by virtue of its simplicity, it may be used to approximate data in many practical cases.

The constants in this equation have been shown to depend on the past history of the adsorbent[14] and, in a review, Harris [15] mentions an approximate method for the solution of an integral equation for adsorption on heterogeneous surfaces, and also that the constant n has the value $\frac{T}{T_K}$ for $T < T_K$ and the value $n = 1$ for $T > T_K$ where T_K is the temperature at which adsorption changes from capillary condensation to molecular adsorption.

Langmuir[16] considered that an adsorbed gas or vapour formed a unimolecular layer on the adsorbent surface. It was also assumed that the tendency to accumulate more molecules on the surface decreased as the surface became covered and hence that a greater partial pressure was required as the adsorption proceeded.

The equation was of the form

$$x = \frac{ab\, p^*}{1 + a\, p^*} \quad \ldots\ldots\ldots\ldots (9)$$

in which a and b are empirical constants whose values depend on the system under consideration.

Langmuir showed that this correlation gave better agreement than the Freundlich equation for the adsorption of gases at low pressures, but there is a significant divergence at high pressures. The Polyani theory suggested that the layer of adsorbed vapours was many molecules thick, but Carver[17] claimed that this was due to surface roughness.

Capillary condensation

The capillary condensation theory has also been considered by many workers. This theory depends on the fact that surface tension effects cause a liquid held in a fine capillary to have a lower vapour pressure than a large surface of the liquid at the same pressure. This could cause a liquid to appear in surface pores and be in equilibrium with a vapour whose vapour pressure is below that of the liquid in bulk at the same temperature. The increase in capacity of an adsorbent with increasing partial pressure of the vapour may be explained by considering that surface pores vary in size and that the larger pores are only completely filled when the partial pressure is increased.

This form of adsorption cannot occur above the critical temperature of a gas or on flat surfaces. It has been shown[18] that adsorbed gases behave similarly above and below their critical temperatures.

The adsorption isotherms for gases near their condensation points can be divided into two regions, one concave and the other convex to the pressure axis. The high-pressure convex part has been explained both by capillary condensation and by the formation of multimolecular layers. Mantell[2] gives a review of the work of several investigators concerned with this theory to cover all five types of adsorption. The theory has been shown to be more generally applicable than the capillary condensation theory[13] in that it includes unimolecular adsorption of the Langmuir type as a special case, the adsorption of many layers on free surfaces and the increase in the amount of adsorption which occurs in capillaries.

An equation was developed[19] which applied to multilayer adsorption

$$\frac{p^*}{v}(P_S - p^*) = \frac{1}{V_m C} + \left[\frac{(C-1)}{V_m C}\right]\frac{p^*}{P_S} \quad \ldots\ldots (10)$$

in which P_S is the saturated vapour pressure of adsorbate at the adsorption temperature, V_m is the monolayer concentration of adsorbate and C is a constant and is the difference

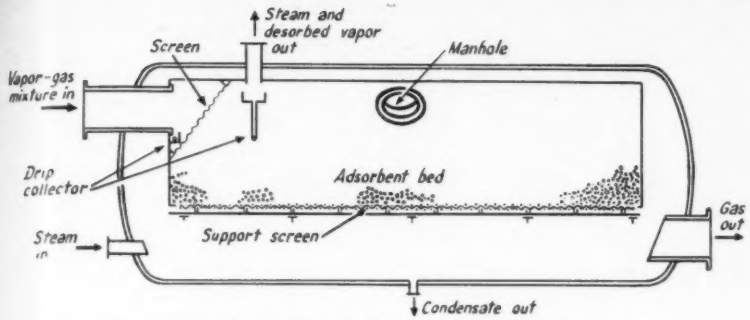

Fig. 5. Schematic adsorber for solvent vapours.
(*U.S. Pat.* 2,186,712)

between the heats of adsorption and liquefaction of the adsorbate.

Factors have been proposed[20] to correct equation (10) for the decrease in pore diameter with increasing adsorption and to allow for the difference in free energy of the second and higher layers. The equation has also been derived statistically by several methods. Harkins and Stearns[21] have proposed another form for the adsorption isotherm

$$-RT\ln\frac{p^*}{P_S} = U_m - \frac{RT}{n}\ln\frac{\theta}{1-W\theta} \quad \ldots\ldots\ldots (11)$$

in which $\frac{p^*}{P_S}$ is the relative pressure, θ is the relative surface coverage and W, n and U_m are constants.

Adsorption in solvent recovery

When air containing solvent vapours is passed through activated carbon almost complete removal of the vapour from air streams containing low concentrations of the solvent may be obtained. Vapour recoveries of up to 99.8% have been reported[22] from air streams containing less than 0.5% by volume of vapour.[23] An adsorption system may be used following a condensation or absorption system, in which the bulk of the vapour has been removed, and give considerable gains.

In order to obtain a vapour-free air stream, adsorbents are usually present in excess and any approach to equilibrium is avoided, except where fractionation of two materials is required. In the design of adsorbers it is usually the times of the adsorbing and desorbing cycles that determine the amount of carbon to be used as well as the dry gas capacity, which is the volume of gas that may be passed through the adsorber before the concentration of vapour reaches an unacceptable level.

In a typical adsorber (Fig. 5), taken from Treybal,[24] the bed of carbon is large in cross-sectional area and shallow, in order to reduce pressure drop through the unit. The gas velocity is usually low and, in order to reduce abrasion of the granular bed, it has been suggested[25] that the maximum safe gas rate is given by

$$G^2 = 0.0167\, D\, g\, d_g\, d_a \quad \ldots\ldots (12)$$

in which d_a and d_g are the apparent bed density and gas density, D is the mean particle diameter, and the constant was found by experiment on full-size plant, using upward flow of air.

Frequently the gas stream must be filtered to remove suspended particles and also it may be necessary to cool it.

Following the adsorbing cycle the bed is desorbed with steam and the solvent is separated out by decanting or distilling. At the start of the next cycle the adsorbed water is either removed with hot air or is allowed to be desorbed by the solvent vapour, which may be preferentially adsorbed.

In some cases two adsorbers may be needed if it is found that a curve of vapour concentration in effluent gas *versus* effluent volume is not sufficiently steep, giving a vapour concentration which begins to rise, in the effluent, while much of the adsorbent is still unsaturated. This curve is known as the break-through curve.

Extensive reviews of the apparatus used in solvent-recovery operations are given by Mantell,[2] Benson and Courouleau[14] and Robinson.[26] A plant to handle very large volumes of air has been described by Luaces.[27]

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Work of the Patent Office

The Comptroller-General's annual report on the work of the Patent Office in 1959 was published in June. The report refers to the continued increase in applications for patents and trade-marks and in applications from other countries, including the Soviet Union.

The continued rise in applications and the difficulty in recruiting staff have led to a substantial arrear of unexamined patent specifications, although this has not increased in proportion to the number of applications.

The report refers to those fields in which new development has been most marked. Applications for trade-marks continue to increase, partly it is thought because of the increased advertising of consumer goods and a tendency to seek a trade-mark for each new line rather than a mark covering a whole range of goods.

Mention is made of the committee which is considering the law relating to industrial designs and of international discussions in the field of industrial property.

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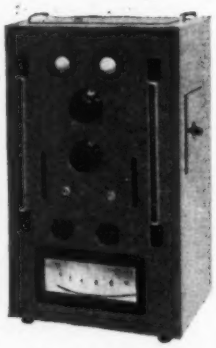

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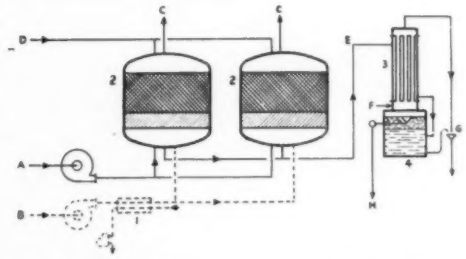

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Design of a Continuous Counter-current Adsorber

By F. Molyneux,* Ph.D., B.Sc.(Lond.), B.Sc.(Eng.), A.M.I.Mech.E., A.M.I.Chem.E.

Mass-transfer operations for solid-fluid systems include adsorption, drying and leaching. Whereas drying and leaching have been extensively studied as chemical engineering unit operations, adsorption has so far received inadequate attention. In this article the author discusses adsorption as a unit operation in order to design from basic principles a continuous counter-current adsorber for removing impurities from dry-cleaning solvents. This original approach should prove particularly useful in developing continuous solvent recovery techniques.

THE removal of colours and impurities in dry solvent liquors is becoming of increasing industrial importance, and in this connection a considerable quantity of carbon tetrachloride contaminated with elementary iodine was available from the solvent extraction of iodine in water with CCl_4. It was found impossible to remove and recover the elementary iodine by any other technique but that of adsorption and, on examination of the literature and some experimental tests, it was decided that the most suitable adsorbent for the purpose was active granular magnesium oxide. This adsorption was carried out isothermally at a temperature of 25°C.

Active magnesium oxide has found application as an adsorbent in the treatment of gasoline and the regeneration of used dry-cleaning solvent. In the first application it removes H_2S and converts mercaptans to H_2S and organic sulphides removing the H_2S formed.

Its use in the regeneration of dry-cleaning solvent rests upon the fact that it adsorbs most polar compounds and so removes colour acids and other materials that tend to give the solvent an unpleasant odour and make it unfit for use.

The solvent, after filtration, is passed through towers containing granular adsorptive MgO. Minor uses of MgO have been in the adsorption of small quantities of materials such as some of the vitamins which are subsequently recovered by washing the oxide with a highly polar solvent.

Magnesium oxide, which has been used for the adsorption of colouring matter from non-aqueous solvents, can be regenerated by treating with clean solvent containing 1 to 5% ethanol methanol, ethyl ether or other strongly polar non-aqueous substances, which later is removed by evaporation in dry air at a temperature not exceeding 100°C.

The following figures were obtained on analysis of activated granular magnesia:[4]

SeO_2	= 1.7%	CaO	= 2.1%
Fe_2O_3	= 0.3%	MgO	= 95.7%
Al_2O_3	= 0.2%	Loss on ignition, 7 to 8%	

Design problem

Ten gal./min. of carbon tetrachloride at 25°C. carrying 1% W/W of elementary iodine is to be scrubbed with granular magnesium oxide to reduce the iodine content to 0.01% W/W. This operation is to be carried out isothermally and counter-currently with granular-activated magnesium oxide. Determine the dimensions of the column to be used.

Theoretical considerations

If L = Mass velocity of adsorbate-free adsorbent
V = Mass velocity of solute-free fluid
X = Mass ratio of adsorbate to adsorbent in L stream
Y = Mass ratio of solute to solute-free fluid in V stream

a solute material balance over the tower is

$$V(Y_1 - Y_2) = L(X_1 - X_2)$$

This gives the equation of the operating line on XY co-ordinates as a straight line of slope L adjoining the terminal conditions $(X_1\ Y_1)$ and $(X_2\ Y_2)$.

An equilibrium curve for the system at this temperature is plotted on the same co-ordinates falling below the operating line.

As for absorbers, the minimum solid-fluid ratio is given by the operating line of maximum slope which anywhere touches this curve and an operating figure of 1.25/2.0 of this is used.

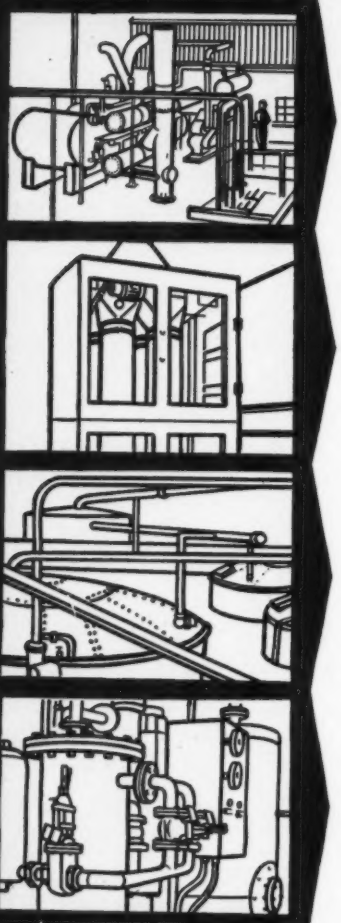
The resistance to mass transfer of solute from the fluid to the adsorbed state on the solid will include that residing in the fluid surrounding the solid particles, that corresponding to the diffusion of the solute through the fluid within the pores of the solid, and possibly an additional resistance at the time of adsorption. During physical adsorption the last of these will probably be negligible.

If the remaining resistances be characterised by an overall fluid mass-transfer coefficient Ka_p based on a_p, the outside surface of the solid particles, then the rate of solute transfer over the differential height of adsorber dZ may be written:

$$L\,dX = V\,dY = Ka_p\,(Y - Y^*)\,dZ$$

where Y^* is the equilibrium composition of the gas corresponding to the adsorbate composition X.

*Senior Lecturer, Department of Chemical Engineering, Birkenhead College of Technology.



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The driving force $Y - Y^*$ is then represented by the vertical distance between operating line and equilibrium curve.

Rearranging and integrating the number of transfer units N_{ov} can be defined by:

$$N_{ov} = \int_{\frac{1}{2}}^{Y_1} \frac{dY}{Y - Y^*} = \frac{Ka_p\, dZ}{V} = \frac{Z}{H_{tov}}$$

This integral is obtained graphically and the packed height Z determined from the overall height of a transfer unit H_{tov}.

From the experimental data of Ahlberg[2] on the adsorption of water vapour from air by silica gel, Hougen and Marshall[3] found that the overall height of a transfer unit could be expressed by:

$$H_{tov} = \frac{1.42}{a_p}\left(\frac{D_p\, G}{\mu}\right)^{0.51}$$

This correlation, modified in the usual manner for liquids, gives:

$$H_{tov} = \frac{1.42}{a_e}\left(\frac{D_p\, G}{\mu}\right)^{0.51}\left(\frac{\mu}{\rho D_L}\right)^{0.67}$$

where a_e = effective surface area of solid particle
= average value = 0.3 a_p
D_L = diffusion coefficient of iodine in CCl_4
= 0.000062 sq.ft./hr.

Properties of activated granular magnesia

Mesh size		D_p *ft.*	a_p *sq.ft./sq.ft.*
100%	20 to 28	0.00226	1,140

Density of granular MgO = 25 lb./cu.ft.
Density CCl_4 entering = 98 lb./cu.ft.

Table 2

Y	$Y - Y^*$	$\frac{1}{Y - Y^*}$
0.0135	0.01140	87.7
0.0102	0.00920	108.8
0.0068	0.00623	160.5
0.0034	0.00311	321.5
0.0017	0.00155	645.2
0.0010	0.00092	1,088.0

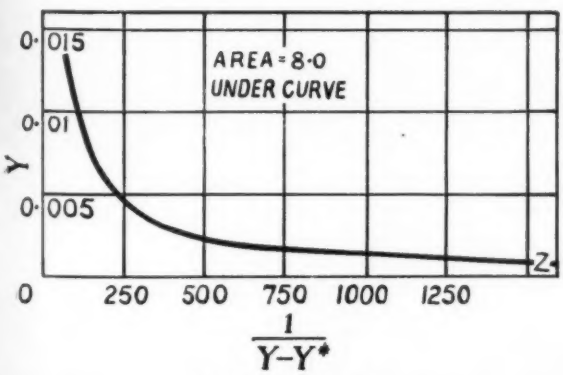

Fig. 2. Integral Curve.

Calculations

The min. $\frac{L}{V}$ from the graph = 1.

Using as the operating slope $\frac{L}{V}$ = 1.25.

$V = 10 \times 15 = 150$ lb./min.

L = operating flow = 150 × 1.25 = 190 lb./min.

By a material balance the iodine content of the exit magnesia stream = [0.01 – 0.0001] (1.25)
= 0.01238 lb. I_2/lb. MgO

The operating line can be drawn through the points (0.01, 0.01238) and (0.0001, 0.0), see Table 1 and Fig. 1.

Plotting Y against $\frac{1}{Y - Y^*}$ for graphical integration, see Table 2 and Fig. 2.

Table I. Equilibrium data iodine/CCl_4/MgO[1] (*See Fig. 1*)

Y $\frac{g.\ solute\ (I_2)}{g.\ solvent\ (CCl_4)}$	X $\frac{g.\ solute\ (I_2)}{g.\ adsorbent\ (MgO)}$
0.00068	0.0101
0.00169	0.0152
0.00254	0.0178
0.00338	0.0190
0.00507	0.0203
0.01350	0.0228
0.00068	0.0101

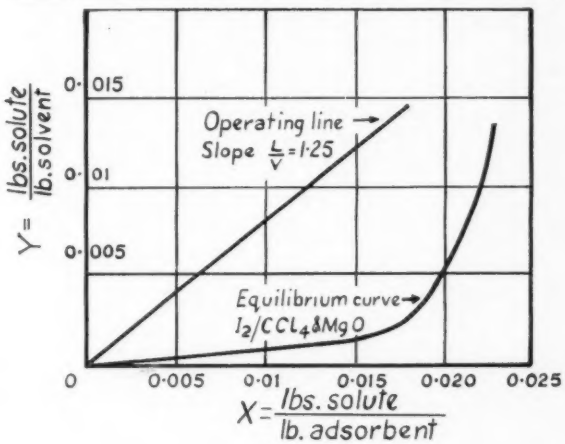

Fig. 1.

Integrating graphically
Number of transfer units = 8.0
Flow of liquid = 10 gal./min.

At an equivalent diameter of 6 in. for the adsorber the fluid velocity = 8.2 ft./min.

D_p = 0.00226 ft.

$$a_e = \frac{1{,}140 \times 30}{100} = 340 \text{ sq.ft./cu.ft.}$$

$G = 8.2 \times 98 = 804$ lb./sq.ft. min.

μ for CCl_4 at 25°C. = 1 centipoise

$$\left(\frac{D_pG}{\mu}\right) = \frac{(0.00226)\,(804)}{0.0672} = 27.0$$

$$\left(\frac{D_pG}{\mu}\right)^{0.51} = (27.0)^{0.51} = 5.36$$

$$\left(\frac{\mu}{\rho D_L}\right) = \frac{0.0672}{(98)\ (0.000062)} = 11.1$$

$$(11.1)^{0.67} = 5.0$$

$$H_{tov} = \frac{1.42}{340}\ (5.36)\ (5.0)$$

$$= 0.112 \text{ ft.} = 1.35 \text{ in.}$$

Packed height = 8.0 × 1.35 = 10.5 in.

Allowing a thickness of adsorbent of ½ in./bed gives 21 beds.

At 3 in. between beds allowing 7½ in. for end connections, overall dimensions of adsorber = 6 ft. 6 in. tall.

Adsorption tower diameter

The only suitable correlation for these conditions which would be discovered in the literature was the old Colburn flooding data for packed extraction columns (Fig. 3).

The later work of Pratt has shown that below a packing size of ½ in. the flow pattern is independent of packing size and it can therefore be assumed that these curves might be used as a guide in the present case.

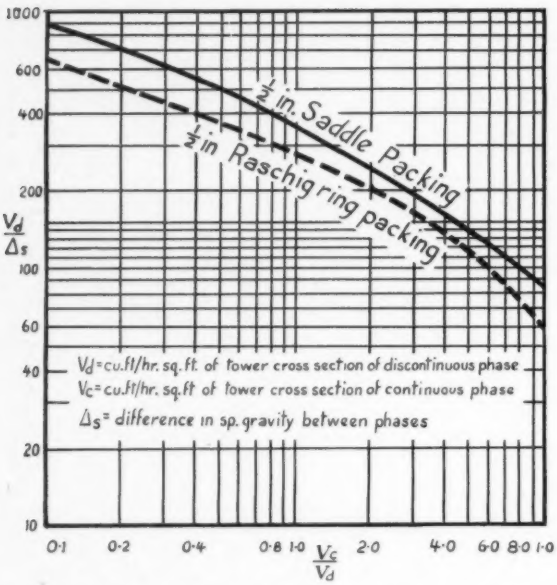

Fig. 3. Colburn flooding data for packed extraction columns.

Flow rates are as follows:

Carbon tetrachloride flow rate = 150 lb./min.

Magnesia flow rate = 190 lb./min.

Specific gravity of carbon tetrachloride = 1.5

Specific gravity of magnesia = 0.4

Then V_d = Velocity of dispersed phase MgO =

$$\frac{190}{0.4 \times 62.4} = 7.65 \text{ cu.ft./min.}$$

V_c = Velocity of continuous phase =

$$\frac{150}{1.5 \times 62.4} = 1.6 \text{ cu.ft./min.}$$

$$\frac{V_c}{V_d} = \frac{1.6}{7.65} = 0.21$$

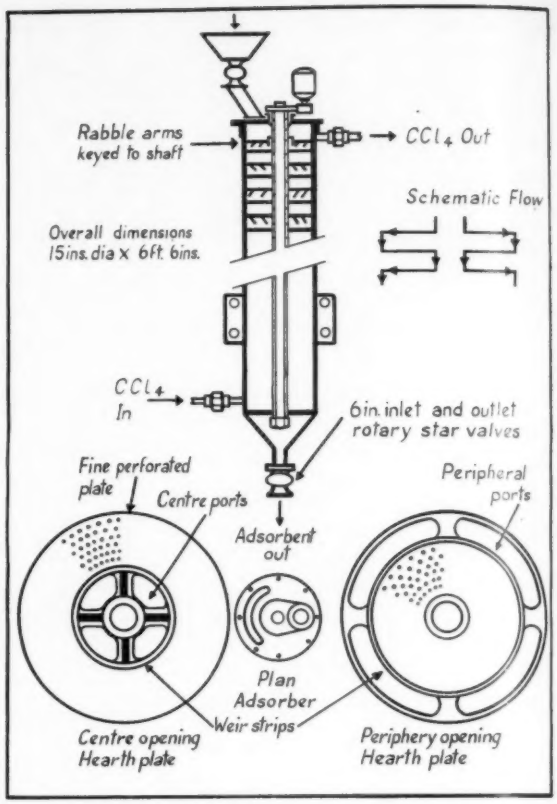

Fig. 4. Continuous counter-current adsorber.

From Fig. 3: $\frac{V_d}{\Delta s}$ = 510 cu.ft./hr. sq.ft.

$$\Delta s = (1.5 - 0.4) = 1.1$$

$$V_d = \frac{1.1 \times 510}{60} = 9.35 \text{ cu.ft./min. sq.ft.}$$

Using the accepted figure for loading of 70 to 80% of flooding gives an operating velocity: 9.35 × 0.7 = 6.56. Then operating empty tower cross-section =

$$\frac{7.65}{6.56} = 1.23 \text{ sq.ft.}$$

$$\text{Diam. } D = \frac{\sqrt{1.23 \times 4}}{\pi} = 1.22 \text{ ft.}$$

Let adsorption tower diam. = 1 ft. 3 in.

Then overall dimensions are 15 in. diam. × 6 ft. 6 in. tall and the percentage free area on each plate to give a fluid flow of 8.2 ft./min. is 25%.

This 25% free area on a 15-in.-diam. column requires ports on each plate to give an area equivalent to that of a 6-in.-diam. pipe.

Construction of plant

The body of the adsorber consisted of one piece of solid drawn mild-steel pipe 15 ft. nominal bore × ¼ ft. wall × 6 ft. 6 in. overall length.

The top of the vessel is made from a standard blank flange with a central gland to take the rabble shaft.

The base of the vessel is conical, converging to a 6-in. off-take fitted with a rotary valve for spent oxide take-off.

The design of the adsorber is based on that of the multiple-hearth furnace such as the *Nichols Herreshoff* and *Wedge* furnaces used for roasting ores.

The granules of magnesium oxide are slowly rabbled across each hearth, dropping from one to the other in counter-current flow to the CCl_4 liquid.

The granules are fed at the top of the adsorber and taken off from the conical base through liquid-tight rotary feed valves.

For regeneration and recovery of the elementary iodine, the shell is wrapped with a copper coil close-pitched and brazed to the adsorber shell. During the adsorption cooling water is passed through this coil and during regeneration steam is passed through the coil and the iodine purged out of the magnesia by means of a current of hot air blown through the adsorber counter-current to a flow of spent granules.

The iodine vapour can be condensed and recovered as crystal in a tubular condenser.

Some difficulty was experienced with lubricants for use with carbon tetrachloride and, in fact, the only success was obtained with a sample of fluorocarbon grease developed for atomic energy production.

During the course of the experimental work on various commercial adsorbents, a sample of commercial anhydrous sodium sulphite *ex* synthetic phenol manufacture was tried on this system.

The adsorption on a batch trial was remarkably good and the capacity quite high.

No equilibrium data could, however, be discovered on the system $I_2/CCl_4/Na_2SO_3$ in the literature, but in view of the capacity revealed, further work is proceeding.

The elementary iodine can be recovered easily by a simple treatment with commercial B.O.V. and condensation of the liberated iodine in a tubular condenser.

The sodium sulphite used was anhydrous, containing 1 to 2% moisture contaminated with sodium phenate (0.5%). Screen analysis: 20 mesh, 10%; 50 mesh, 65%; and 100 mesh, 25%.

At the present price of commercial sodium sulphite of this grade (£11 to £12/ton) present indications of adsorptive capacity indicate that it might be a cheaper medium than MgO for plant use.

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INDUSTRIAL SOLVENT RECOVERY

Static Process

IN industries employing organic solvents economic operation depends to a great extent on the efficient recovery of solvent, ensuring its continuous utilisation in the process and thereby preventing its evaporation to waste. It is also of vital importance that only that solvent which is best suited to a particular process—not necessarily the cheapest—should be selected because this to a large extent influences the quality of the product and ensures the best operating conditions whereby personnel are safeguarded against the effects of noxious vapours.

The *Supersorbon* process, manufactured under licence by Chemical Engineering Wiltons Ltd., is based on utilising the physical surface forces of highly activated carbon. This carbon is manufactured by a special activating method and has a micro-capillary structure, giving an extremely large surface area of about 600 to 1,200 sq.m./g. Various brands of this carbon are comprised of cylindrically shaped granules of 1 to 4 mm. which totally adsorb even small traces of organic solvents from air or gases within a few seconds. The solvent is easily desorbed from the carbon by application of steam and can be recovered in its original strength and purity.

Operation

The method of operation of such a plant may be seen from the diagram (Fig. 1). Solvent-laden air (A) is collected by means of a suction fan (1) from the area where evaporation occurs and is delivered to the adsorbers

1 – suction-air fan
2 – drying air and cooling air fan (necessary only for special solvents)
3 – airheater (necessary only for special solvents)
4 – adsorber
5 – condenser
6 – separator

A – solvent laden air
B – drying and cooling air
C – solvent free air
D – rinsing steam
E – distillate
F – cooling water
G – waste water
H – solvent

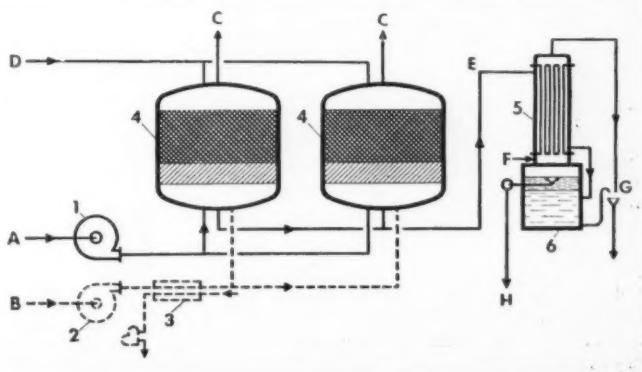


Fig. 1.

(4) which are filled with activated carbon. This process continues until the carbon is fully saturated with solvent. This condition may be easily determined by a slight smell of solvent emerging from the exhaust of the first adsorber, when the adsorber is disconnected and another brought into service. Changing adsorbers is best done automatically by a concentration analyser in conjunction with power-operating changeover valves.

Concentration analyser

The *Solvomat* concentration analyser is particularly useful where solvent concentration varies, and measures the solvent concentration present in the exhaust gases (C) and automatically triggers the switching of adsorbers to desorption as soon as they have become saturated with solvent. By preventing adsorbers from becoming overcharged or undercharged before desorption, wastage of solvent and steam is prevented.

Steam desorption

Reconditioning of a saturated adsorber is done by passing steam through it in a counter-current direction (D). The recovered solvent is contained in the condensate and is afterwards mechanically separated. If the solvent is completely or partially soluble in water, the condensate must be treated either in a rectification unit or in a unit combining condensation with distillation. Such a unit presents a combined process utilising the waste energy of the reconditioning system.

In the case of many solvents a bed of heat economiser stones, located beneath the carbon bed, may be employed to recover from the outgoing desorbing steam, heat which is stored and later used to heat the incoming solvent-laden air which dries the carbon bed. After the heat in the stones has been dissipated the continued flow of air cools the carbon bed.

As can be seen only three operating stages are required—adsorption, desorption and separation of solvent from condensate. No additional equipment is required other than a supply of steam which may be flash generated.

Inherent safety of the process permits recovery of even highly inflammable solvents such as carbon disulphide, with absolute safety. For each pound of solvent recovered the requirements are 2 to 3½ lb. of steam, 3 to 5 gal. of cooling water and 0.1 to 0.15 kWh. of electricity. The activated carbon has many years of useful life and depreciation of the mechanical plant is very low.

Continuous Plant

A continuous solvent recovery unit has been designed and developed by Sutcliffe, Speakman & Co. Ltd. specifically to meet modern demands for equipment which is continuous and automatic in operation. The solvent-laden air is initially filtered to remove any fluff or dust particles and then cooled or heated to the appropriate temperature before it reaches the adsorber. Only one adsorber is used. This consists of a horizontal rotor, containing an annular bed of active carbon divided into sections, which rotates slowly and continuously. While the adsorber rotates and other sections of the carbon bed are adsorbing solvent, simultaneously and without arresting the motion of the adsorber, each segment in turn passes through a positively segregated zone in which steam is automatically passed through this isolated carbon bed (see Fig. 2).

The released solvent in vapour form and the steam is directed to the condenser and the condensate passed to a seal tank. In the case of non-water-miscible solvents, this tank is adapted to act as a separator and continuously divides the condensate into a solvent layer and a water layer which leave the unit separately by gravity flow.

Some solvents, however, although virtually non-water-miscible, retain in solution small percentages of water, *e.g.* ethyl acetate. In these instances, if a completely dry solvent is necessary for the process, the water content is removed by treating the solvent solution in a dehydrator using a hygroscopic adsorbent.

When the solvent or solvents are water-miscible, the condensate is taken to a distillation column for final separation. This distillation plant can be arranged for separation of water-solvent systems, and for the separation of solvent mixtures. The unit can be designed for automatic control or for manual operation and arranged for continuous or for batch system working. All the equipment necessary for the solvent recovery process, together with the adsorber, rotating gear and the filter-cleaning apparatus, is housed within the one main casing. Noteworthy features of the continuous solvent recovery plant are:

(*a*) The continuous cycle of adsorption and release of the solvent from the carbon.
(*b*) Only one adsorber necessary for the process.
(*c*) An even consumption of steam and water—no peak demands.
(*d*) No cyclic-operation valves.
(*e*) The large amount of work done by the comparatively small quantity of carbon.
(*f*) The very limited amount of floor space occupied.
(*g*) All parts are readily accessible and the main casing access doors are fitted with quick-release fasteners.
(*h*) The plant can be delivered as a complete unit and installed immediately on a previously prepared flat concrete base.

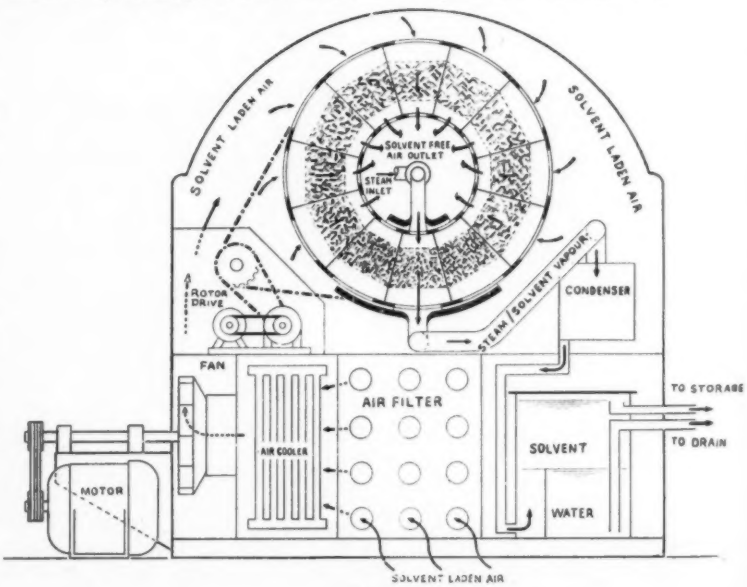


Fig. 2.

Uranium Dioxide Fabrication

By F. S. Martin,* M.Sc., Ph.D., A.R.C.S., F.R.I.C., and B. R. Steele,* Ph.D.

The tendency for reactor technologists to favour non-metallic fuels and thus to turn from metallurgy to chemistry is one of the most significant features in the modern nuclear industry. Uranium dioxide is one of the most promising chemical fuels and the preparation, fabrication and properties of this compound are described in this article.

IN his search for nuclear reactor fuels which have greater stability and longer life than uranium and its metallic alloys, the nuclear fuel technologist is turning increasingly from metals to non-metallic compounds.

Chemistry plays a significant role at almost every stage of ceramic fuel manufacturing processes, and chemical reactions serve in large part to explain the behaviour of fuel within the nuclear reactor. The compounds which the fuel technologist is interested in are, naturally, those of the metallic elements which already serve as fertile and fissile fuels, *i.e.* compounds of thorium, uranium and plutonium. A short list of these with examples of their applications are shown in Table 1.

Table 1. Some non-metallic solid nuclear fuels

Fertile or Fissile element	*Compound (pure or mixed)*	*Type of reactor use*	*Example*	*Remarks*
Th	ThO_2	Breeder blanket	LMFR (Brookhaven, U.S.A.)	Solid fuel or aqueous slurry
U	UO_2 UC UO_2/Inert metal UO_2/Inert compound USi	Thermal	PWR, AGR UO_2/Fe; UO_2/Al UO_2/Be	Also as blanket for enriched U thermal reactors or Pu fast reactors
Pu	PuO_2 PuC	Fast or thermal spike		
U, Th	UC/ThC/C UO_2/ThO_2 UO_2/Glass	Thermal breeder	DRAGON ZENITH	High-temperature gas-cooled type
U, Pu	UO_2/PuO_2	Fast or thermal spike		

Preparative methods

One of the most promising uranium compounds in the table which can be used as a high-temperature fuel is uranium dioxide. For use as a fuel it is frequently required as fabricated shapes of as near theoretical density as possible. Because of its very high melting point (2,850°C.), fabrication by melting and casting is not feasible. Instead, the qualities that are required of UO_2, and indeed of most solid non-metallic fuels, are conveniently obtained by using powder processing methods, *e.g.* for uranium dioxide a 'surface reactive' powder is produced by a series of chemical operations which after compaction is sintered to a high-density product. Similarly, uranium carbide may be prepared by allowing the elements to react under the right conditions in a suitably prepared powder form.

The reactions occurring during compacting and sintering involve to a large extent surface properties. The surface properties of many powders are significantly dependent upon their physico-chemical history and therefore the chemical techniques used for preparing feed materials for ceramic nuclear fuel fabrication processes have a pronounced influence on the quality of the product.

The usual starting material for the large-scale production of uranium dioxide powder is uranyl nitrate and there are two general methods which can be used for the conversion to uranium dioxide:

(1) By thermal denitration to uranium trioxide and subsequent reduction,

(2) By precipitation from solution as uranium peroxide or ammonium diuranate, followed by thermal decomposition to a higher oxide and its subsequent reduction.

The powders obtained from these two routes differ markedly (see below).

Thermally denitrated powder

Uranyl nitrate solutions from ore extraction processes are concentrated by evaporation to approximately the hexahydrate composition (*i.e.* $UO_2(NO_3)_2.6H_2O$).

The thermal denitration of uranyl nitrate hexahydrate can be carried out in pots or in continuously operated equipment. At the Mallinkrodt chemical works in the U.S.A. batch denitration is carried out in gas-heated stainless-steel pots of 40- to 250-gal. capacity.[1] The denitration cycle is shorter in the smaller-capacity batches and correspondingly affects the properties of the resultant trioxide. In particular, the smaller batches give a greater proportion of orthorhombic UO_3 which is less reactive than other forms of UO_3. Denitration can also be carried out continuously by spraying concentrated uranyl nitrate solution into a fluidised bed of UO_3 particles at 300 to 350°C. The UO_3 is reduced to dioxide using either hydrogen or cracked ammonia gas at temperatures from 400 to 800°C.

* *Development and Engineering Group, U.K.A.E.A., Springfields, Lancs.*

Microstructures of UO_3 and UO_2 particles prepared by the fluidised-bed technique are shown in Figs. 1 and 2; the particles are approximately spherical and have a porous internal structure. Some of the larger particles are formed by agglomeration of smaller ones during the denitration operation.

The surface area of these UO_2 particles, as measured by the B.E.T. inert gas adsorption technique, lies in the range 1 to 5 sq.m./g. It depends upon that of the starting UO_3 and on the reduction temperature. For instance, if the reduction of the UO_3 is carried out at 500°C., the surface area of the UO_2 is approximately equal to that of the UO_3, but higher reduction temperatures lead to sintering and a reduction in surface area.

Precipitation methods

Uranium can be precipitated as uranium peroxide, UO_4, or as ammonium diuranate (ADU) by the addition of hydrogen peroxide or ammonia to uranyl nitrate solution. Different precipitation conditions lead to precipitates of widely differing characteristics. The precipitation of uranium peroxide is quite a specific reaction for uranium so that it can also be used as a purification step, whereas precipitation with ammonia gives little decontamination from common metallic impurities.

Peroxide precipitation

Uranium peroxide can be precipitated from hot uranyl nitrate solution by the addition of hydrogen peroxide at a *p*H as low as 1.5; an 'inoculation' technique is used for the production of a very uniform precipitate.[2] The UO_4 precipitate is fine grained with approximately spherical particles; on further simultaneous addition of peroxide and nitrate solution, the particles grow but still retain their spherical shape. A small quantity of the precipitate is used to inoculate subsequent precipitations, which are carried out using increasingly more concentrated reagent solutions. By careful control of the procedure, a very uniform precipitate of constant particle size is obtained.

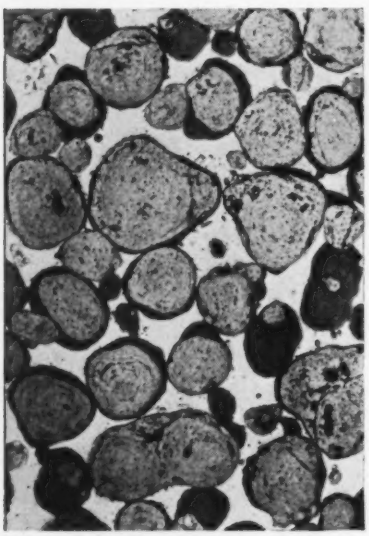


Fig. 1. Uranium trioxide (UO_3) prepared from thermally denitrated uranyl nitrate.

Ammonium-diuranate precipitation

The effects of the following variables in precipitation are important:

(i) Concentrations of uranyl nitrate and ammonia;
(ii) Rate of reagent addition and mixing;
(iii) Temperature; and
(iv) Final *p*H of solution.

Small particle precipitates are obtained by rapid precipitation with the addition of excess concentrated ammonia to dilute uranium solutions.

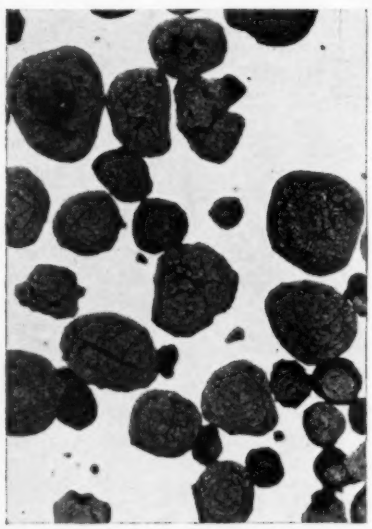


Fig. 2. Uranium dioxide (UO_2) as prepared by hydrogen reduction of thermally denitrated trioxide.

In batch-type precipitation the conditions vary throughout the experiment; the *p*H of the solution increases and the uranium concentration decreases. This variation naturally leads to the formation of a wide range of irregular shaped particles (Fig. 3). A more uniform precipitate can be obtained by homogeneous precipitation, *e.g.* by the decomposition of urea in boiling solution. ADU crystals obtained in this way are more uniform in size and roughly spherical in shape. Continuous precipitation, for example, by feeding the two reactants continuously into a suitable vessel where rapid mixing takes place, also leads to a more uniform product and eliminates batch-to-batch variations.

Particle characteristics, *e.g.* shape and size, are preserved throughout the sequence of reactions

$$ADU \xrightarrow{\text{heat}} UO_3 \xrightarrow{\text{heat}} U_3O_8 \xrightarrow{H_2} UO_2$$

Fig. 3. So-called ammonium di-uranate precipitate.

and it is the precipitation step which largely determines the final size, size distribution and shape of the UO_2 powder particles. A typical UO_2 powder, produced from batch precipitated ADU, is shown in Fig. 4.

The conditions used for the thermal decomposition and hydrogen reduction reactions control to a large extent the size of the crystallites (as distinct from particles) in the UO_2 powder and its surface area as measured by inert gas adsorption. At relatively low temperatures (400 to 500°C.) the UO_2 powder has approximately the same surface area as the starting material; as the decomposition and reduction temperatures are increased, the crystallite size increases until the powder particles are virtually single crystals.

UO_2 powders of high surface area (*e.g.* about 8 sq.m./g.) oxidise readily in air at room temperature to compositions as high in oxygen as $UO_{2.2}$. This oxidation can proceed so rapidly

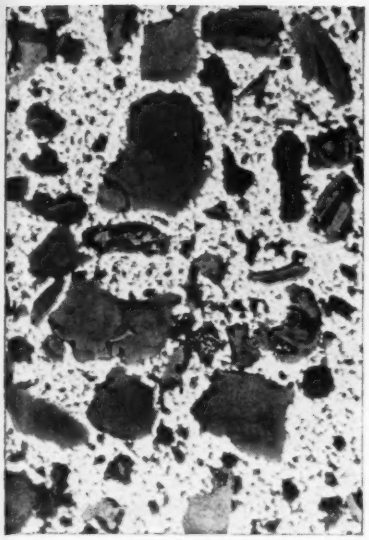


Fig. 4. Uranium dioxide prepared by hydrogen reduction of pyrolised ammonium di-uranate precipitate.

that the heat generated by the oxidation can cause the powder to reach as high a temperature as 500 to 600°C., reconverting the UO_2 to U_3O_8. Lower surface area powders (*e.g.* 2 to 3 sq.m./g.) are stable in air, but oxygen is chemisorbed on the surfaces to give average compositions corresponding approximately to the formula $UO_{2.04}$.

Fabrication methods

Given a suitable feed powder, a number of chemical and mechanical operations are then involved in its conversion to a ceramic product. The mechanical operations are those which may be encountered in both chemical and metallurgical processing industries. Stages such as blending, mixing and granulation use familiar equipment, whilst a stage such as compaction will require the use of a press akin in potential output to those used for pharmaceutical tablet production on one hand and on the other hand to those used for precision compacts in powder metallurgy, *e.g.* tungsten carbide products. The chemical stages are naturally peculiar to whatever type of powder is being handled and are confined usually to reactions with binding agents, with ambient atmospheres and to physico-chemical surface or solid-state reactions associated with sintering. These stages are exemplified by the flow diagrams shown in Fig. 5 and which refer to processes of the type used for uranium dioxide fuel manufacture. The significance of each stage is briefly considered in the following paragraphs.

Granulating aids may fall into either of the two fields, chemical or mechanical. Mechanical treatments may involve milling, which serves to 'de-air' the powder and to modify surface properties and to change the powder aggregate 'shape' so as to facilitate the clumping together of aggregates to form approximately spherical granules. Chemical aids may either modify powder surface properties, *e.g.* by the adsorption of small amounts of moisture which greatly facilitates 'self-granulation', or they may act as macroscopic binding agents by forming a matrix between the powder particles. In the latter case the binder is usually a high-molecular-weight organic polymer (*e.g.* polyvinyl alcohol or polybutylmethacrylate or a wax) used as a solution in water or in an organic solvent.

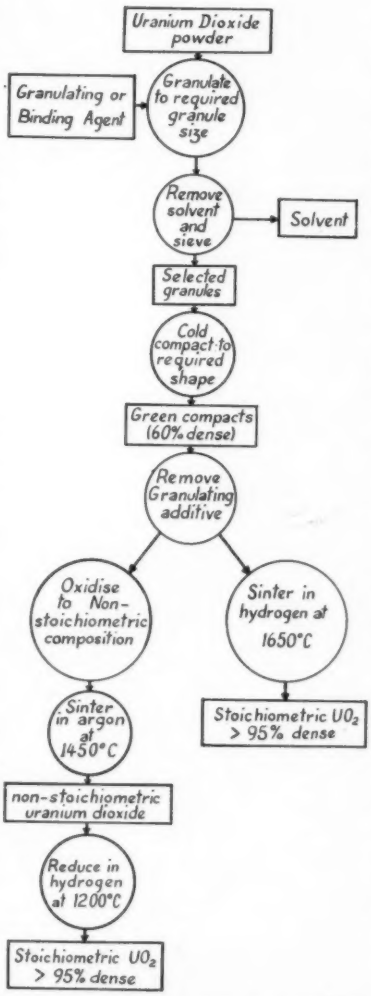


Fig. 5. Typical flow-sheet for uranium dioxide fuel production.

Granulation achieves a number of objects and is becoming increasingly important as a process stage in many industrial operations.[3] In the present case granulation ensures a smooth, even-flowing feed to automatic pressing equipment; it reduces dust formation and dissemination and hence also reduces toxic ingestion hazards. Where the powder has been activated by the addition of trace quantities of water, granulation is simply achieved by tumbling the powder in rubber-lined drums when it readily aggregates into roughly spherical shapes, the required granule size range being subsequently selected by sieving. If an organic binder is used, sufficient is added to the powder to make a plastic mass suitable for granule formation by crude extrusion through an appropriate sieve. Before these granules are used for pressing, any solvent which may have been used as medium for the binder is evaporated. At this stage also, lubricants (usually organic fats or waxes) may be added as finely divided solids or as solutions in appropriate solvents, the latter, of course, being removed prior to the pressing or compacting operation.

Compaction produces solids of the required shape from the granular feed. In general, the quality of compacts, *i.e.* perfection of surface, freedom from voids and laminations, is improved where binding agents and particularly lubricants have been used. If neither of these has been used, it is frequently advantageous to lubricate those surfaces of the dies and punches which come into contact with the powder.

Removal of granulating additive or lubricants

Lubricants may be based on glycerol tristearate and are usually present in relatively small proportions, *e.g.* 0.1% by weight; binding agents are usually polymeric organic compounds and are present to the extent of a few per cent. by weight. All of these additives tend to decompose on heating, leaving a carbonaceous residue. At the high temperatures involved in the sintering operation (*e.g.* about 1,650°C.) any carbon residues in the UO_2 compact may react to form uranium carbide. Unfortunately, uranium carbide is readily hydrolysed by moisture and forms hydrocarbon gases which cause disintegration of the pellet. It is thus usually necessary to remove the organic material before the sintering stage.

If water has been used as the granulating addition and the compact contains only a small quantity of added

lubricant, a separate removal stage prior to sintering is not usually necessary. Most active UO_2 powders usually contain excess oxygen to about the composition $UO_{2.04}$ (equivalent to about 0.25% by weight of oxygen in excess of that required by the stoichiometric formula). This additional oxygen is adequate to ensure the removal of any traces of carbon formed by the decomposition of the 0.1% of lubricant. If all the carbon in the lubricant is removed as carbon dioxide, 0.1% of lubricant requires 0.2% of oxygen. If an organic compound has been added as the binding agent, then up to about 3% of carbon could be formed if none of the carbon in the polymer were lost during its decomposition.

Since the amount of non-stoichiometric oxygen present in UO_2 powder, as normally prepared, is not sufficient to remove this quantity of carbon, a separate binder removal stage is incorporated in the fabrication sequence. The carbon cannot be completely removed by oxidation in air, as at the required temperatures, the UO_2 compacts would disintegrate owing to the formation of the less dense U_3O_8. Atmospheres with lower oxidation potentials may, however, be used which will oxidise the carbon but not the UO_2. For instance, treatment of 'bonded' UO_2 compacts at 800°C. with carbon dioxide removes the carbon completely and rapidly by the reaction:

$$C + CO_2 \longrightarrow 2CO$$

The oxidation of UO_2 in CO_2 at 800°C. is negligible and the carbon monoxide formed ensures that the pellets are of near stoichiometric composition after this treatment. Alternatively, one may use wet hydrogen, in which case the carbon is removed by the water-gas reaction:

$$C + H_2O \longrightarrow CO + H_2$$

The carbon monoxide and hydrogen again maintain the stoichiometric composition in the compacted oxide.

Sintering of UO_2

Cold compacted UO_2 shapes have densities in the range 5.5 to 7 g./c.c., *i.e.* 50 to 60% theoretical density, depending on the compacting pressure used and the characteristics of the feed UO_2 powder. The final densification to 95% theoretical density and greater is achieved by heating these compacts to temperatures at which the rates of diffusion and plastic flow are sufficiently great to achieve this density in a relatively short time. At high temperatures there is a considerable solubility of oxygen ions in UO_2; the UO_{2+x} phase has considerably different physical properties from those of $UO_{2.0}$. In particular, it has a lower viscosity at a given temperature, so that densification proceeds at a much faster rate in non-stoichiometric oxide than in the stoichiometric compound at the same temperature.[4] In fact, non-stoichiometric oxide can be sintered to 95% theoretical density at temperatures of less than 1,400°C., whereas the sintering temperature for the stoichiometric material is about 1,600 to 1,700°C.

This lower sintering temperature leads to a wider choice of furnace design and a longer and less exacting life for the furnace refractories. However, the furnace has to operate in an inert atmosphere (argon or nitrogen), as any reducing gases would rapidly remove the non-stoichiometric oxygen and prevent sintering; an oxidising atmosphere would convert the whole charge to U_3O_8.

The preparation of non-stoichiometric oxide for these sintering operations can be achieved in several ways. Firstly, a very reactive powder may be used which oxidises on exposure to air to a composition $UO_{2.1}$ to $UO_{2.2}$; this method, however, is difficult to control. Secondly, a controlled oxidation of a less reactive UO_2 powder may be achieved by restricting the quantity of available air. A third and more satisfactory method is to mix UO_2 and U_3O_8 in the correct proportions and to allow these to react during the heating period of the sintering operation to form non-stoichiometric UO_2.

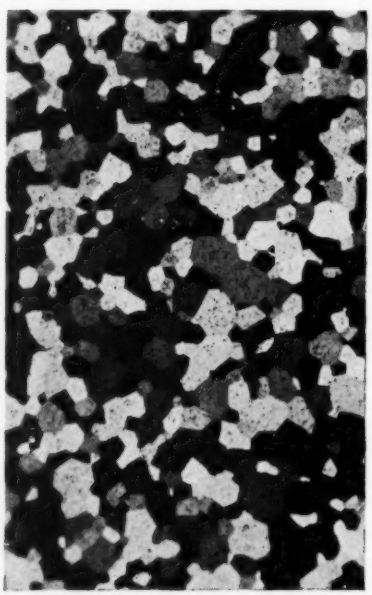


Fig. 6. Photomicrograph of sintered uranium dioxide showing grain structure.

The incorporation of an organic binding agent in a non-stoichiometric sintering route may raise difficult problems. The decomposition of small quantities of lubricating material will not materially affect the non-stoichiometry of the compact, but the use of larger quantities of organic polymeric material as binding agent may reduce the non-stoichiometric oxygen. This may be reintroduced by using a slightly oxidising atmosphere in the sintering furnace, *e.g.* steam or carbon dioxide. The dissociation of steam at 1,400°C. is sufficient to oxidise $UO_{2.0}$ to $UO_{2.17}$ which will sinter at this temperature. Oxidation to this composition is possible with carbon dioxide, but the reaction is much slower and relatively long sintering times are required.

Sintering of stoichiometric oxide is carried out in a reducing atmosphere of hydrogen or cracked ammonia gas at temperatures of 1,650 to 1,750°C. Molybdenum heating elements are used, the whole furnace being kept in the reducing atmosphere to prevent oxidation of the molybdenum. The heating and cooling periods of batch-type furnaces operating at these temperatures must be quite long; up to 24 hr. for a sintering time of only 1 to 2 hr. to avoid excessive thermal shock of the furnace refractories.

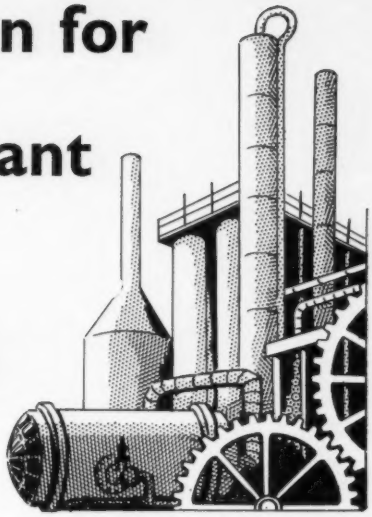
For large-scale production, continuously operated furnaces have obviously a great advantage. Not only are the lengthy heating and cooling times eliminated, but the continuous temperature cycling of the refractories is avoided, thus considerably increasing their life. The heating rate of small UO_2 compacts, *e.g.* cylindrical pellets 0.3 × 0.3 in., can be extremely rapid; samples have been heated from room temperature to 1,650°C. in 1 hr. without any deleterious effects. In a continuously operated furnace of this type elaborate gas locks at the entrance and exit are not required, as the hydrogen flame itself acts as a curtain to prevent any entrance of air to the furnaces.

Properties of sintered UO_2 compacts

The density of a UO_2 compact sintered under fixed conditions (*e.g.* 2 hr. at 1,650°C. in a hydrogen atmosphere) varies considerably with the characteristics of the powder used. Surface-active powders prepared from ammonium diuranate precipitates sinter

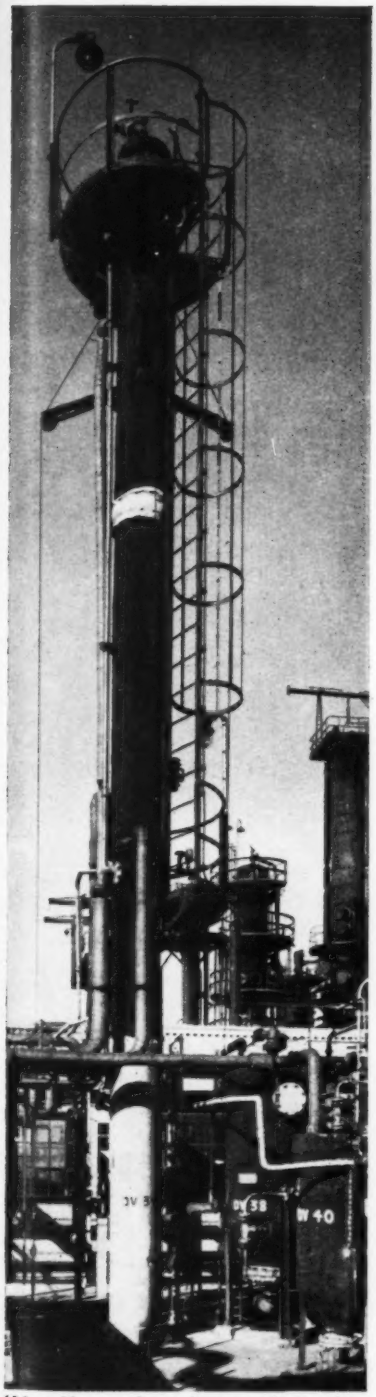
(*Concluded on page* 303)

Materials of Construction for Chemical Plant



NICKEL

By J. Hinde*
F.I.M., M.Inst.W.



'Monel' caustic stripper column for mercaptan extraction at the refinery of Lobitos Oilfields Ltd.

This is the third article in our series on 'Materials of Construction for Chemical Plant', which has so far included PVC and Lead. Nickel is a very expensive metal, and this factor alone prevents its acceptance as a general-purpose material of construction. The combination of excellent corrosion resistance with high mechanical properties, however, have made nickel and nickel alloys irreplaceable for many process vessels. This authoritative article describes the properties of nickel, Monel, Inconel *and other nickel alloys and discusses the usefulness of these materials to the chemical plant constructor.*

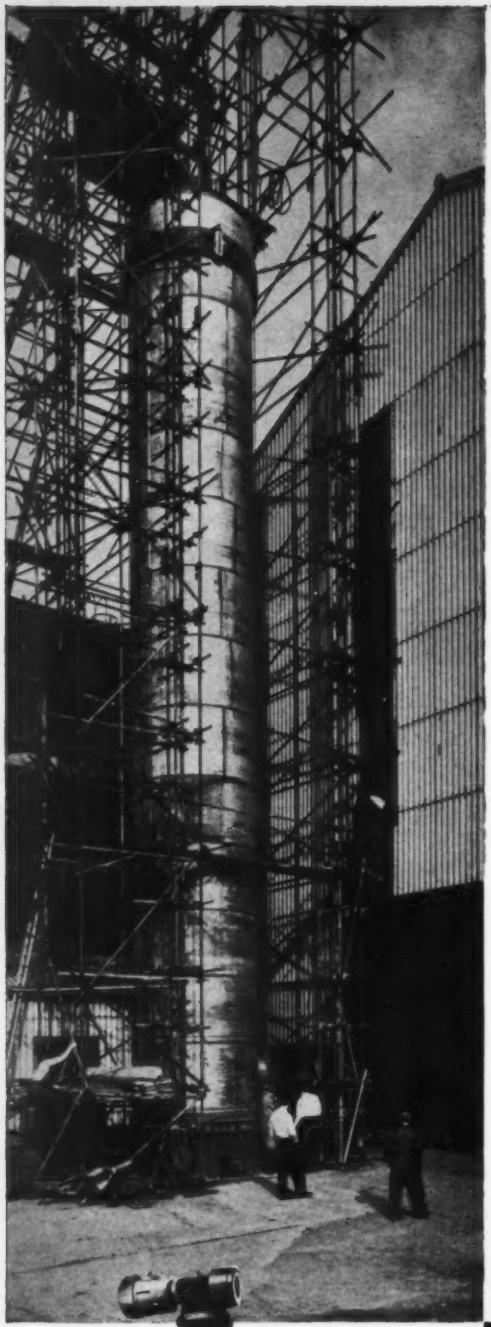
PREHISTORIC man used nickel-iron alloys to tip his weapons thousands of years before Cronstedt isolated the element nickel in 1751 and Bergman described his preparation of metallic nickel in 1775. Man-made alloys of nickel with copper and zinc, smelted from mixed ores, were used by the Chinese, and a Bactrian coin 'minted' about 235 B.C. has been found to contain 78% copper and 20% nickel, a composition closely akin to that of modern 'silver' coinage, which is based on 75% copper, 25% nickel.

The first major use of nickel may be traced to the early 19th century, when Europeans began to make their own varieties of the nickel-copper-zinc alloy called by the Chinese 'paktong', and this alloy still has its counterpart in 'nickel silver'.

Due to its sulphur content, the metallic nickel then available was relatively brittle and it was not until 1879 that this problem was overcome by Fleitmann in Germany, who found that the addition of small amounts of manganese and magnesium yielded a metal which was malleable and ductile. As early as 1843, Boettger had developed a nickel-plating process, but the real value of electro-deposited nickel as a corrosion-resistant surface was established only in 1861, by Adams, and this application has since then remained one of the major outlets for nickel.

By the early 1900s nickel-alloy steels had assumed a commercial significance, a development which was associated with names such as Faraday and Bessemer, but it is the present century which has seen the advent of the great variety of corrosion- and heat-resisting alloys now available.

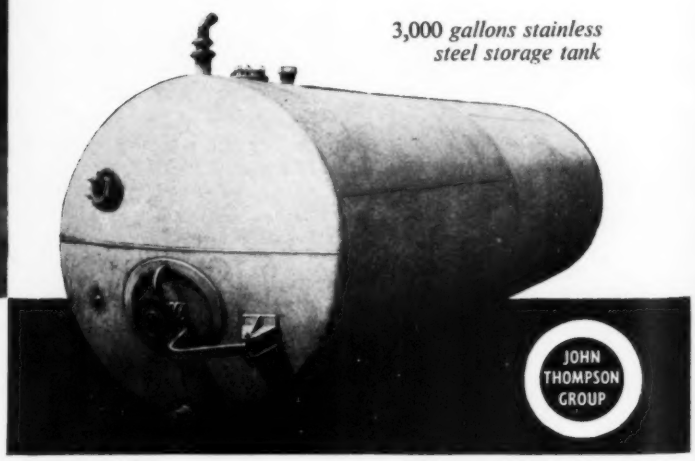
**Research and Development Department, Mond Nickel Co. Ltd.*



Chemical Vessel and Process Plant

John Thompson (Dudley) Ltd. fabricate plant for all industrial chemical processes in mild steel and special materials such as stainless steel, Monel, nickel, Inconel and clad and non-ferrous metals. Fabrications for complete constructions including large pressure vessels and installations are undertaken with pipework chimneys and supporting steelwork.
Thompson Bros. (Bilston) Ltd. manufacture storage and process tanks, vessels and mixers of welded construction in mild steel, stainless steel, aluminium, Monel and other metals to client's exact requirements.

One of six absorption columns for I.C.I.



3,000 *gallons stainless steel storage tank*

1,500 *gallons mild steel jacketed mixer*

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Table I. Composition and properties of nickel and high-nickel alloys in the annealed condition

Material	*Nominal composition % max., unless otherwise stated*									*0.2% Proof stress t.s.i.*	*Max. stress t.s.i.*	*Elong. % on 2 in.*	*Typical hardness values D.P.N.*	*Approx. melting range °C.*	*Specific heat c.g.s. 20 to 100°C.*	*Therm. cond. c.g.s. 20 to 100°C.*	*Coeff. of expan. c.g.s. $=10^{-6}$ 20 to 600°C.*	*Annealing temp. °C.*
	(a) Ni	C	Si	Mn	Fe	Cu	Cr	Mo	Others									
Nickel	*(b)* 99.0 min.	0.15	0.15	0.35	0.4	0.25	—	—	—	8	30	45	110	1,435 to 1,455	0.110	0.145	16.5	815 to 925 Air cool
Nickel-copper alloy	63.0	0.3	0.5	2.0	2.5	28 to 34	—	—	—	12	34	45	135	1,300 to 1,350	0.127	0.062	16	870 to 980 Air cool
Nickel-chromium iron alloy	72.0	0.2	0.5	1.0	10	0.7	14 to 17	—	—	15	40	40	140	1,395 to 1,425	0.109	0.036	15.5	980 to 1,065 Air cool
Nickel-molybdenum alloys	Bal.	0.1	1.0	1.0	5 to 8	0.4	—	26 to 30	*(d)*	28	60	40	250	1,345 to 1,370	0.09	0.027	12	1,150 to 1,175 Rapid cool
Nickel-molybdenum-chromium-alloy	Bal.	0.1	1.0	1.0	4 to 7	—	14.5 to 16.5	15 to 17	3 to 4.5 W	30	60	40	250	1,270 to 1,300	0.092	0.025	14	1,210 to 1,230 Rapid cool
Nickel molybdenum-chromium-iron-alloy *(a)*	40.0	0.05	0.5	1.0	31	1.25	21	3	—	14	40	57	150	1,280 to 1,320	*(c)* 0.10	*(c)* 0.03	*(c)* 17	1,120 to 1,150 Rapid cool
Nickel molybdenum-chromium-iron-alloy *(b)*	45.0	0.05	1.0	2.0	13.5 to 17.5	— —	21 to 23	5.5 to 7.5	1.75 to 2.25 Nb	22	45	46	170	1,270 to 1,300	*(c)* 0.10	*(c)* 0.03	*(c)* 17	1,150 to 1,175 Rapid cool

(a) Includes residual cobalt
(b) Carbon content 0.02% max. in low-carbon quality
(c) Estimated values
(d) Controlled composition + vanadium to avoid heat treatment after welding

One of the first corrosion-resisting alloys was made in 1905 by Stanley of the International Nickel Co. He treated a composite matte—shades of Chinese practice!—to produce directly an alloy containing roughly two-thirds of nickel and one-third of copper. This alloy, much stronger than nickel, was found to resist attack by industrial atmospheres, sea-water and non-oxidising acids, and it soon found commercial applications under the name *Monel*, given to the alloy in honour of Ambrose Monell, then president of Inco. Since then, nickel has been an important constituent of many corrosion-resisting materials, notably the austenitic stainless steels and the cupro-nickels. In addition, several other nickel-base corrosion-resisting alloys have been developed for service in the chemical industry, and it is the properties of these materials which will form the subject of this article. Table 1 lists the nominal compositions and some of the properties of the alloys discussed.

Some idea of the growth of the nickel industry may be gathered from the fact that about 100 years ago the world's total production was around 500,000 lb., whereas the 1960 figure for the free world is expected to exceed 500 million lb. Nickel has been important in peace time and 'strategic' in war: during the 1939-45 and the Korean War periods end-uses were restricted and, indeed, the metal remained in short supply until quite recently. The now rapidly increasing rate of production will, however, ensure supplies adequate to meet all demands.

Corrosion resistance

Commercial nickel has useful resistance to a wide range of corrosive media,[1, 2, 3] but modern use of the metal for chemical-process equipment is based essentially on its particular resistance to the following conditions:

(*a*) Dry chlorine and bromine, over a wide range of temperatures.
(*b*) Chlorinated solvents which, as the results of hydrolysis, may contain small concentrations of hydrochloric acid.
(*c*) Hot, wet organic chlorides.
(*d*) Hot aqueous solutions of organic neutral and acid chloride salts, excluding oxidising salts.
(*e*) Fluorine at elevated temperatures.
(*f*) Molten salts such as sodium chloride, sodium nitrate and potassium chloride.
(*g*) Molten sodium and potassium.
(*h*) Caustic soda, at all temperatures and concentrations.

A major use of nickel is in plant for the concentration of caustic alkalis. The resistance of nickel to highly concentrated caustic soda or to the fused alkali results from the protective nature of the oxide film which forms on the surface of the metal. In the presence of this thin black film the rate of corrosion by fused caustic soda is of the order of only 0.003 in. p.a. Temperatures above 320°C. favour the development of the film, but when handling molten caustic soda at such temperatures it is necessary to use a special grade of low-carbon nickel (0.02% carbon); this material resists the intergranular attack which may develop in nickel of the more usual carbon content (around 0.05%).

In some instances the choice of nickel, as of many other materials, is based not so much on resistance to corrosion in terms of weight-loss as upon the ability of the metal to prevent discoloration or contamination of the products with which it is in contact. For many years the rayon industry has specified caustic soda of very low iron content, and to ensure that degree of purity nickel and nickel-clad steel have been widely used for handling alkali of that grade. An unusual example of this application is afforded by the use of nickel-clad steel for the holds of the S.S. *Dow Chem*, a vessel which for some years past has regu-

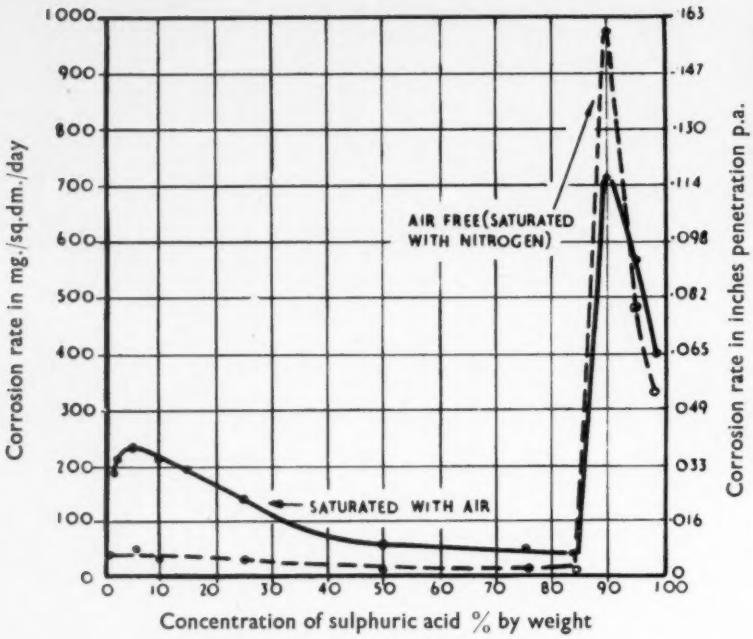

Corrosion rate in mg./sq.dm./day
Corrosion rate in inches penetration p.a.
AIR FREE(SATURATED WITH NITROGEN)
SATURATED WITH AIR
Concentration of sulphuric acid % by weight

Fig. 3. Iso-corrosion curves for 'Monel' in sulphuric acid.

Fig. 1. Hold in S.S. 'Dow Chem', fabricated from nickel-clad steels, to carry caustic soda. Pipes and ladder are in solid nickel. The picture illustrates the excellent condition after two years' service.

larly transported high-grade caustic soda. These holds are illustrated in Fig. 1, which shows their excellent condition after some years of service.

Another example of the use of nickel to prevent contamination of product is in the production and handling of water-white phenol. A number of materials show, in phenol, a rate of attack low enough to be of no concern in determining the life of the plant, but where water-white phenol is called for nickel is chosen in order to avoid the pink colour which develops so rapidly in the presence of traces of iron. Fig. 2 illustrates a phenol-transport tanker in which the tank and its heating coils were fabricated throughout in nickel.

Alloys with copper

Mention has already been made of the nickel-copper alloy *Monel* which, because of its high nickel content, is similar to nickel in its resistance to corrosion in the media referred to in the previous section. The resistance of *Monel* to molten salts and alkali metals is inferior to that of nickel, but the copper content imparts superior resistance to non-oxidising chloride solutions and to hot dilute solutions of the non-oxidising acids. Many of the special applications of *Monel* involve the handling of acids such as hydrochloric, sulphuric and phosphoric, and excellent service experience has been recorded with plant used, for the pickling of steel, the production of ammonium sulphate and detergent manufacture involving contact with sulphonated organic compounds.[4–7, 11] Figs. 3 and 4 are iso-corrosion rate curves illustrating the resistance of *Monel* to hydrochloric and sulphuric acids.

In the non-oxidising acids corrosion can be markedly accelerated by the presence of dissolved air or of other oxidising substances which promote cathodic oxidation of hydrogen. This effect of aeration is illustrated in Figs. 3 and 4.

Monel has good resistance to unaerated hydrofluoric-acid solutions, at most concentrations and temperatures.[8] For this reason the alloy is

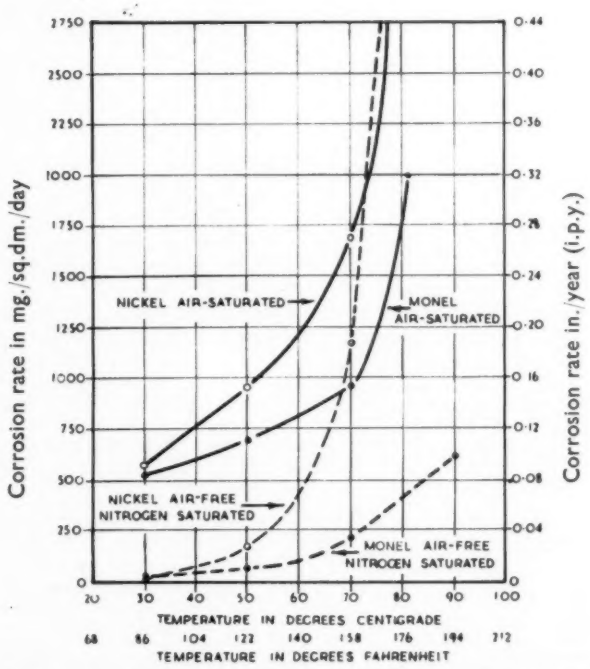


Fig. 4. Iso-corrosion curves for nickel and 'Monel' in hydrochloric acid.

Fig. 2. Road transport tanker in nickel, to carry phenol, showing mounting on chassis.

widely used for the regeneration of hydrofluoric acid in processes involving the use of that acid in the alkylation of the petroleum hydrocarbons[9] (Fig. 5). *Monel* has also found extensive application in plant for the fluorination of uranium oxide, in the processing of nuclear fuel.

This alloy is widely used in plant for the production of salt; tests in hot saturated brine indicate a corrosion loss of only 0.002 i.p.y. Examination of *Monel* drier linings after many years of service has shown that in steam-heated plant the rate of thinning is well below 0.0015 i.p.y. In oil-fired driers the corrosion rate is higher, as would be expected from the greater quantities of salt handled in a given period, but the figure is still less than 0.010 i.p.y., and this includes the additional corrosive effect of sulphur compounds on the fuel side. *Monel* is used also in several types of sea-water evaporators, and other applications include the handling of sodium- and calcium-chloride solutions in refrigerating brines.

Monel is not resistant to the oxidising acid salts nor to acids containing oxidising agents, and caution should be exercised with regard to the alloy in ' chamber-process ' sulphuric acid. Of the oxidising alkaline salts, the hypochlorites are the only ones which seriously attack this, and even in such medial attack occurs only when the available chlorine exceeds 3 g./l. The behaviour of *Monel* in phosphoric acid is greatly affected by the presence of oxidising impurities, notably ferric compounds. In the manufacture of phosphoric acid by the treatment of phosphate salts with sulphuric acid the presence of iron compounds in the product will limit the practical use of *Monel*, but in the phosphorus-vaporisation process, which yields a pure acid, the alloy has proved very useful for equipment in the absorption towers.

Thus the corrosion-resisting properties of *Monel* are such that the material

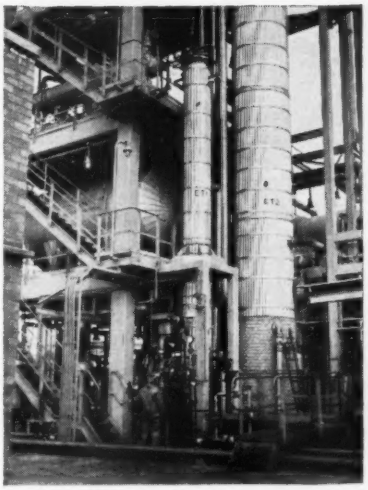


Fig. 5. Hydrofluoric acid regeneration column in ' Monel ' installed at Grange Chemical Ltd.

finds many applications in the chemical and process industries, in power plant and in marine equipment. Its applications in the chemical industries have been detailed in many publications dealing with individual items of equipment, and are well summarised in ' Developments in Nickel ', a review based on a symposium of the Division of Industrial and Engineering Chemistry of the American Chemical Society[10] and in other publications dealing specifically with high-nickel alloys[2] (Fig. 6).

The basic composition of *Monel* in the cast form may be varied by additions of silicon, which greatly increase the wear resistance and anti-galling properties of the alloy. The addition of aluminium to the wrought material imparts age-hardening response and enables *K Monel*, as the aluminium-containing alloy is called, to develop high-tensile properties in conjunction with the corrosion-resisting characteristics of standard *Monel*. *K Monel* is widely used for shafts and engineering details in which advantage can be taken of its particularly good mechanical properties.[12]

Nickel-chromium-iron series

Representative of alloys of this group is *Inconel* (nominal composition 76% nickel and 15% chromium, balance mainly iron). In oxidising conditions its chromium content imparts resistance superior to that of pure nickel or the nickel-copper alloys, whilst its high nickel content maintains a considerable resistance to reducing conditions. In some air-saturated mineral and concentrated organic acids the oxidising effect of air alone will not ensure complete passivity and freedom from attack.

Inconel is one of the few materials resistant to hot, strong solutions of magnesium chlorides; in addition to its low rate of surface corrosion, the

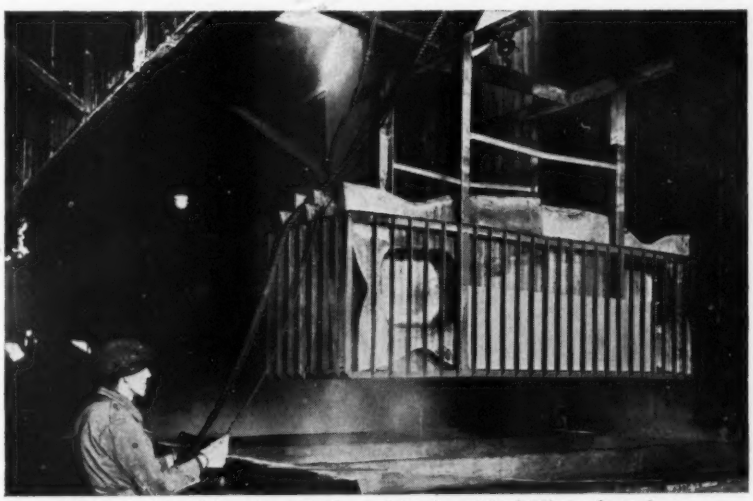
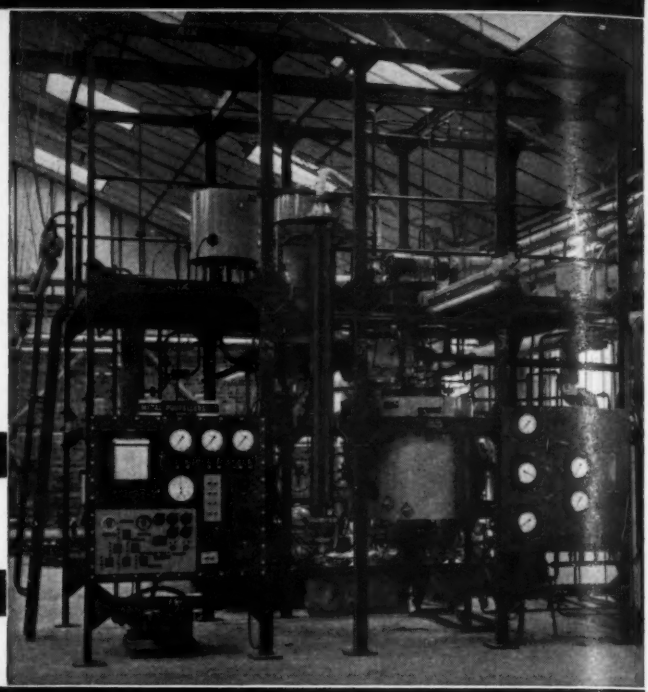


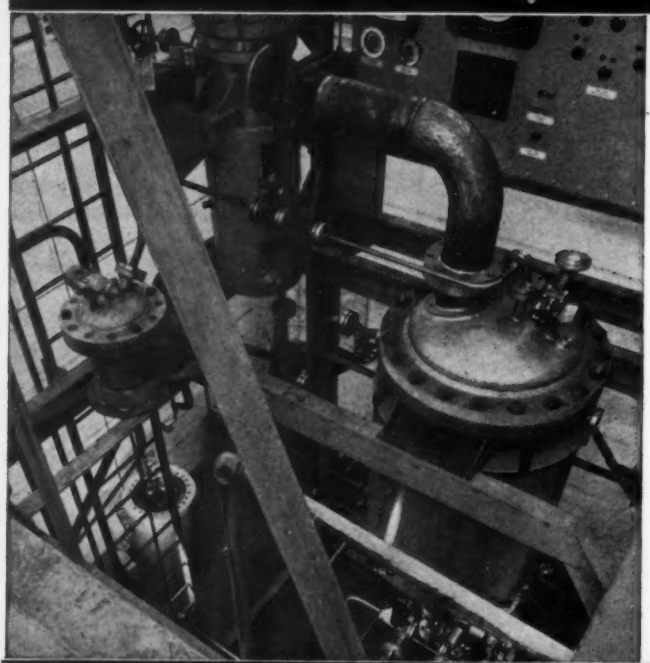
Fig. 6. ' Monel ' crate used for sulphuric acid pickling of steel.

PILOT PLANT FOR THE PETROLEUM & CHEMICAL INDUSTRIES



40-gallon stainless steel general purpose reactor at the Abingdon Laboratories of Esso Research Ltd.

Operating platform view of batch/continuous distillation plants, looking downwards to induction-heated lead bath preheater (left) and multiple pump unit.



We have made a variety of stainless steel pilot scale units from time to time, from simple distillation plants, or reaction vessels, of 1-10 gallons capacity, to complex continuous or batch process plants which provide maximum versatility and flexibility combined with comprehensive instrumentation and control gear, and reproduce full scale methods.

The two versatile plants illustrated here were designed from flow data as packaged units for hydrocarbons research and development work, and embodied full instrumentation and control gear and electric heating.

All process vessels, etc., are in stainless steel to B.S.S. EN.58J (316), thus ensuring optimum corrosion resistance to a wide range of possible products, maximum range of operating temperatures, and freedom from product contamination.

We shall welcome the opportunity of co-operating with you on the design of any pilot plants you may be requiring.

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alloy shows no tendency towards stress-corrosion cracking during exposure to such solutions. This resistance to stress-corrosion cracking is a general attribute of the high-nickel alloys and, because of this characteristic, *Inconel* has proved a most useful material in the pressurised-water type of nuclear reactor.

In its resistance to oxidising acid salts *Inconel* is superior to nickel or *Monel*, but it is still not immune to attack by ferric, cupric and mercuric chlorides. It is virtually unaffected by silver-nitrate solutions and finds extensive application in the manufacture of photographic chemicals and in film processing equipment.

Inconel has no particular merit for service in sulphuric and hydrochloric acids, but it shows useful resistance to strongly oxidising acids, including nitric acid in concentrations above 20%. More dilute solutions of the acid may, however, cause significant attack. In general, the stainless steels are a more economic proposition than *Inconel* for nitric-acid service, but it is interesting to record the recent development of an alloy based on 65% nickel, 35% chromium, to give resistance to hot, concentrated nitric acid in conditions for which the stainless steels are not satisfactory.

fluoride (Fig. 7). In the process industries *Inconel* finds many applications, *e.g.* in production of plastics and in the paper industry. In the form of cladding on steel plate, or as weld deposits overlaid on steel, *Inconel* has been used to fabricate sulphate pulp digesters, in which service it has proved very satisfactory.

In addition to its virtues as a corrosion-resisting material, *Inconel* has also considerable merit as a heat-resisting alloy. In the exhaust manifolds of piston aero engines it had an excellent service record, and it is still used as the metallic covering of asbestos heat-insulating blankets. In this country the modern jet engines make extensive use of its 'cousins', the *Nimonic* series of creep-resisting alloys, which were developed from the 80% nickel-20% chromium composition. In the U.S.A., developments of *Inconel* involving the addition of titanium and other elements have resulted in creep-resisting alloys such as *Inconel X*, which have also found many applications, but these are mostly in gas-turbine engines and other fields of service which are outside the context of this article. *Inconel* itself still retains many applications in furnace equipment and steam-power plant (Fig. 8).

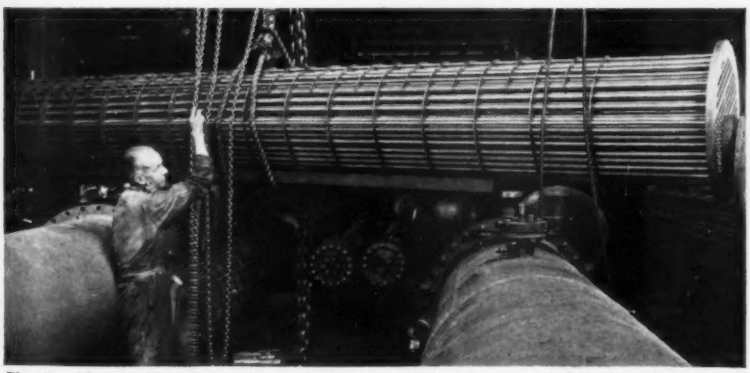


Fig. 8. 'Inconel' tubular heat exchanger of the type used for oil refinery and tar distillation.

Because of the high nickel content, *Inconel* is particularly suitable for handling alkaline solutions; it is especially useful in that it resists attack by alkaline sulphur compounds, which gives the alloy particular merit for plant used in the manufacture of sulphate or kraft paper.

The resistance of *Inconel* to hot, dry gases is a valuable attribute, of which advantage is taken in many applications;[2] these include the handling of anhydrous hydrofluoric acid at elevated temperatures in the preparation of uranium hexafluoride and aluminium

Nickel-molybdenum alloys

Of the other materials listed in Table 1, the nickel-molybdenum-base alloys, which include *Hastelloy B* and *Corronel* 210, may be regarded as extending the corrosion resistance of nickel-base materials to an even wider range of media, concentrations and temperatures. Hydrochloric acid is probably the most corrosive of the common acids; it will destroy protective oxide films and, since chlorides are appreciably soluble, there is little possibility of providing a resistant coating. Only a noble material—one

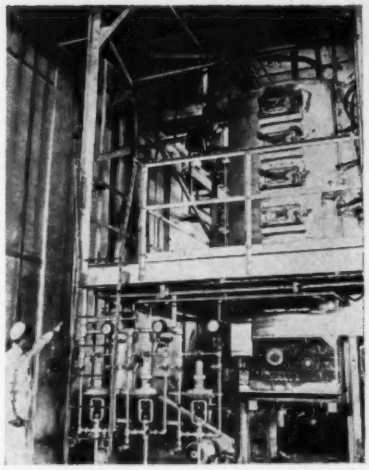


Fig. 7. Plant in 'Inconel' for the production of uranium hexafluoride.

which does not readily release hydrogen from the acid—is likely to offer useful resistance to attack. Copper might be successful, but for the fact that its nobility is reduced by the tendency to formation of cupric-chloride ions and by the marked acceleration of attack which occurs in the presence of oxygen or oxidising agents. Nickel itself possesses a useful resistance to hydrochloric acid, but the value of copper in improving the nobility of nickel is seen in the still better resistance of the nickel-copper alloy to hydrochloric acids in moderate concentrations and under non-oxidising conditions.

Molybdenum has a marked effect on the nobility of nickel and its resistance to hydrochloric acid, and these alloys are often employed where hot and concentrated solutions of hydrochloric acid have to be handled. Most of the commercial alloys based on the nickel-molybdenum system contain 26 to 30% of molybdenum, usually with about 5% of iron, derived from the ferro-molybdenum used in making the alloy. Fig. 9 shows that these materials possess a reasonable degree of resistance to corrosion by the acid over the whole range of temperatures and concentrations usually encountered in service, but in the presence of cupric and ferric salts rate of attack by hydrochloric acid will be greatly accelerated. The nickel-molybdenum alloys are characterised also by good resistance to attack by sulphuric and phosphoric acids (Fig. 10).

The nickel-molybdenum alloys may be readily welded by the fusion-welding techniques usually employed in the fabrication of chemical plant, although in oxy-acetylene welding

there is a risk of carbon pick-up, which will adversely affect the corrosion resistance of the weld deposit. A more serious consideration is, however, the fact that, whatever the method used, welding introduces a risk of severe intergranular corrosion in a zone adjacent to the weld when it is exposed to aggressive media such as hot concentrated hydrochloric acid, under conditions of aeration.

This susceptibility to intergranular corrosion arises from solution of carbides during the welding operation and their re-precipitation, at grain boundaries, on cooling from the welding temperature. This form of attack can be prevented by a post-welding heat treatment, but, since the treatment involves rapid cooling from around 1,180°C., there are obvious difficulties in applying it to fabricated chemical plant. A low carbon content will minimise the effect, but work in the research laboratories of the Mond Nickel Co. has led to a more satisfactory method of eliminating susceptibility. A new alloy, known as *Corronel* 220, has been developed by controlling the limits of the basis composition and adding vanadium. Apart from the value of such a material for chemical plant construction, its use makes possible the lining of vessels, which would be impracticable if heat treatment after welding were required.

Other developments in the nickel-molybdenum alloys have included, notably, an alloy in which about half the molybdenum is replaced by chromium, and tungsten is added. *Hastelloy C*, an alloy of this class, resists wet chlorine up to about 60°C., and strong hypochlorites, ferric chloride and other severe corrosive conditions to a slightly high temperature, under conditions in which no other wrought metallic materials except tantalum and titanium are likely to be serviceable. For this reason the modified nickel-molybdenum-base alloy is used in plant for a variety of wet chlorinations at moderate temperatures.[13] There are also other more complex variations of nickel-base alloys containing molybdenum and chromium, which have been designed to resist specific oxidising acid solutions such as mixtures of nitric with chromic or sulphuric acids and complex sulphate solutions of the type used in viscose-rayon-spinning baths. Such complex alloys are usually in the cast form, and are used mainly for pumps and agitators; alloys typical of this series are *Durco D*. 10, *Illium*, *LaBour* 55 and *Langley* 7*R*.

Another cast nickel alloy of particular interest is the nickel-silicon type containing about 10% silicon. This material has useful resistance to boiling sulphuric acid of most concentrations, and cast coils of nickel-silicon alloy are used for concentrating the acid by evaporation in conditions in which other materials usually considered are the high-silicon irons or tantalum.

Molybdenum-chromium-iron alloys

As will be seen from Table 1, alloys of this group are somewhat lower in nickel content than those previously discussed, and contain substantial amounts of iron, but they can still be classed as nickel-base compositions. *Ni-O-Nel* and *Hastelloy F* are commercial examples of this type of alloy, which may be regarded as a further development of the more highly alloyed austenitic chromium - nickel stainless steels containing additions such as molybdenum and copper. Increasing the nickel content of a chromium-nickel austenitic stainless steel containing both molybdenum and copper increases resistance not only to pitting but also to the stress-corrosion cracking to which the austenitic steels are susceptible. Oxidising agents have a beneficial effect on the general corrosion resistance of this type of alloy, but the presence of reducing agents tends to break down the passive film, although not so readily as in the case of stainless steels.

The nickel-molybdenum-chromium-iron type of alloy has advantages over the stainless steels, and also over *Monel*, in hot sulphuric-acid solutions, whether oxidising agents are present or not. At room temperatures they show good resistance to sulphuric acid at all concentrations, and they are useful at concentrations of about 70% up to a temperature of 80°C. and at 40% concentration up to boiling point. The resistance of these materials to hot sulphurous acid solutions makes them suitable for components handling combustion gases and fuel condensates. They also resist attack by nitric acid but, in general, show no advantage in this connection over the stainless steels unless the nitric acid is present in admixture with another acid, *e.g.* nitro-sulphuric and nitro-hydrofluoric acid solutions.

Of the alloys which have been discussed, all except the cast materials must usually be fabricated into the vessels and other components required in chemical plant. In many cases this involves welded construction, and it is an advantage that all of the wrought

The material of construction for chemical plant which will be discussed in next month's issue of CHEMICAL & PROCESS ENGINEERING will be

STAINLESS STEEL

Of interest to our readers . . .

A number of articles appearing in our associate journals this month will appeal to readers of CHEMICAL & PROCESS ENGINEERING.

Manufacturing Chemist—Evaporators and Evaporation, by P. J. King.

Food Manufacture — Special feature on Quick Freezing and Refrigeration.

Automation Progress — The British Computer Industry Today, by P. M. J. Williamson. Automatic Inspection Devices, by D. Foster.

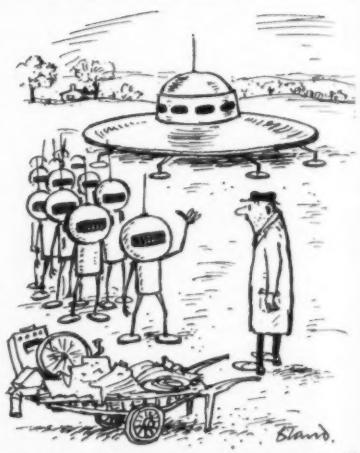
Paint Manufacture—Equipment for the Manufacture of Emulsions, by A. P. Bowden. A comparative study of polyvinyl acetate, co-polymers and acrylics in emulsion paints.

Fibres and Plastics—Analysis and Testing of Plastics, by G. C. Ives.

Petroleum—Special feature on Pipelines.

Corrosion Technology—Stainless Steels in Chemical Industry, by Dr. P. Buchmann. Inhibitors for Aluminium in Acid Solutions, by J. Sundararajan and T. L. Rama Char.

COMICAL ENGINEERING CORNER



'Never mind where we got it, what'll you give us for it?'

alloys under consideration can be welded by the inert-gas processes or by the metallic-arc method. It is not possible to discuss here the details of welding, but it should be emphasised that, since it is important that the corrosion resistance of the welded joints should match that of the parent material, it is vital to use the filler wires or electrodes recommended by the producers of the respective alloys and to follow their advice on welding procedure. There is ample service experience to indicate that welds of appropriate composition, sound and free from faults such as entrapped slag or undercutting, will be satisfactorily resistant to corrosion.

Many of the alloys discussed are relatively expensive, but often they are the only available materials which can be used in a particular chemical process. In some instances the cost of

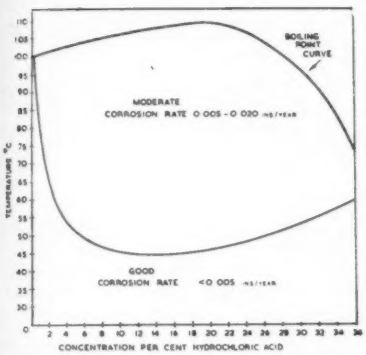

BOILING POINT CURVE
MODERATE CORROSION RATE 0·005 - 0·020 mg/cm²/year
GOOD CORROSION RATE <0·005 mg/cm²/year
TEMPERATURE °C
CONCENTRATION PER CENT HYDROCHLORIC ACID

Fig. 9. Iso-corrosion curve for nickel-molybdenum alloys in hydrochloric acid.

such materials in massive form would be prohibitive and, in such circumstances, it may be possible to use them as a cladding for steels. These clad materials, which comprise mild steel on to which has been rolled the alloy material, require special welding techniques, in order to ensure continuity of the cladding.[14] In some instances strip-lining of the vessel may be practised, *i.e.* individual strips of the corrosion-resisting alloy are welded on to mild steel; yet another method is by an overlay technique, whereby the steel is covered with a layer of the corrosion-resisting alloy by a fusion-welding process. The application of such overlays is most useful on tube plates and flange ends, but the procedure involved can be mechanised to permit treatment of the inside of small autoclaves, etc., of mild steel, thus obviating the use of the alloy material in the very heavy section which would be required to resist the pressure conditions.

Fig. 10. 'Corronel 210' nickel-molybdenum alloy equipment for pickling 'Nimonic' alloys in 20% hydrochloric acid at 110°C.

Particular applications

Monel is one of the oldest of the alloys used in chemical plant and was also the corrosion-resisting material first used for household and decorative purposes. Most of the latter applications have been lost to the stainless steels, but, as already indicated, the nickel-copper alloy still maintains an important place in chemical, power and marine engineering. Nickel itself remains the material pre-eminently suitable for handling caustic alloys of high concentrations and at elevated temperatures; for this application it has not been seriously threatened by any other materials of construction.

The other 'traditional' high-nickel alloy, *Inconel*, is suitable for many applications in which the stainless steels are chosen because of their economic advantage, but when higher temperatures are involved, *Inconel* comes into its own. There is also considerable overlapping in potential applications of the stainless steels and the nickel - molybdenum - chromium-iron alloys where, as would be expected, the cheaper cost of the former decides the choice, but here again there are applications where only the more highly alloyed material can safely be used.

To meet very severely corrosive conditions the nickel - molybdenum alloys are often the only alternatives to precious metals; the possibility of substituting cheaper alloys for the nickel-molybdenum types will therefore seldom arise.

The writer wishes to thank the directors of the Mond Nickel Co. Ltd. for permission to publish this article.

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Uranium Dioxide Fabrication

(*Concluded from page* 294)

to give specimens of greater than 95% of the theoretical density under those relatively mild conditions. In contrast, the less reactive powders produced by thermal denitration, although packing to give higher densities in the unfired state, may only sinter to 85 to 90% of theoretical density under the above conditions. In specimens denser than about 92% of theoretical, practically all the porosity is of the closed type, *i.e.* there is no access to it when attempting to impregnate the pellet with a liquid.

The grain size of sintered UO_2 compacts is to a certain extent determined by the sintering temperature and duration, but again quite large differences can result from the use of more reactive feed material. A photomicrograph of a typical sintered pellet is shown in Fig. 6.

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Fig. 1. The entrance to the factory at Portishead. On the left are the storage silos.

Phosphorus at Portishead

THE element phosphorus occupies a significant position in the chemical hierarchy. With an atomic number 15 it is situated between silicon (14) and sulphur (16) in Period 3 of the periodic table. Both silicon and to some extent sulphur have tended to form independent chemical disciplines compatible with that of carbon chemistry. This approach has enabled chemists to elucidate the systematic chemical structures of the varied silicon and sulphur compounds. In recent years the existence of a number of homologous series in the chemistry of phosphorus has shown that there is the expectation that some day phosphorus chemistry may be developed into as elegant a discipline as present-day organic chemistry. Phosphorus, however, is not quite as freely available as carbon and for this reason the economic manufacture of this product is of utmost importance, to some extent reflecting the rising standard of living in the community.

Organic sources

Although phosphate fertilisers have been used for over 2,000 years, the element phosphorus was not isolated until 1669 when the well-known alchemist Brand prepared it by evaporating urine and distilling the residue to yield sodium metaphosphate. On further heating with carbon this produced phosphorus. This method, though functional, could scarcely be adapted to mass production. A new, plentiful source of raw material was revealed by the Swedish chemist Gahn in 1769, who showed that phosphorus is an essential constituent of bones.

The industrial revolution in England and the growing needs of the match industry created a large-scale demand for phosphorus. In 1844, Arthur Albright started the first commercial production using bones and bone ash as raw material. After grinding, the bones were treated with sulphuric acid and the orthophosphoric acid formed was separated by filtration.

$$Ca_3(PO_4)_2 + 3H_2SO_4 \longrightarrow 3CaSO_4 + 2H_3PO_4$$

This acid was then evaporated to about one-quarter of its volume and mixed with charcoal and coke. The resulting mixture was heated in retorts to convert the orthophosphoric to metaphosphoric acid which was then reduced by the carbon present

$$4HPO_3 + 12C \longrightarrow P_4 + 12CO + 2H_2$$

The phosphorus was distilled off, condensed under water and subsequently purified.

Nowadays the most important mineral sources of phosphorus are fluorapatites, *e.g.* $3Ca_3(PO_4)_2.CaF_2$, known as phosphate rock. Common impurities in this mineral include calcium and magnesium carbonates, ferric oxide and aluminium oxide. The total world consumption of phosphate rock found in immense deposits in the United States, North Africa and the Middle East was about 32 million tons in 1958.

Present-day manufacture

Albright & Wilson, the company which Arthur Albright founded, are at present the sole producers of phosphorus in Great Britain. Their main phosphorus plant at Portishead is beautifully located near the Bristol Channel. Yellow phosphorus produced at Portishead is distributed to other factories in the company to initiate processes that provide a wide range of products, such as insecticides, plasticisers, flotation reagents and oil additives.

Manufacture at Portishead is by the electrothermal process which was patented in 1888 (the process flow sheet is shown in Fig. 6). The three basic materials used in the process are phosphate rock, silica chippings and anthracite. The reaction is carried out under heat in a furnace according to the following equation:

$$2Ca_3(PO_4)_2 + 6SiO_2 + 10C \longrightarrow CaSiO_3 + P_4 + 10CO$$

Storage

An important part of the factory is the unloading and storage facilities which ensure a continuous supply of new materials for the process. Fig. 2 shows a typical ship carrying phosphate rock from Florida standing in the dock at Portishead. Cargoes of about 6,000 tons are unloaded by pneumatic suction plant and then passed by a conveyor belt to storage silos. Each of the eight storage silos, as shown in Fig. 1, contains 2,200 tons phosphate rock, while stored between the silos are approximately 2,000 tons each of anthracite and silica chippings.

The silica and anthracite drawn from the storage silos are dried in the calciners (Fig. 3) and then passed into intermediate storage. From there the various ingredients are weighed batch by batch to give the correct chemical composition for feeding into the furnaces. Electrically controlled weighing machines are used to weigh batches of 4,500 lb. every 2.5 minutes and these are discharged simultaneously on to conveyor belts leading to storage bins above each furnace.

Furnaces

The raw materials are fed into six furnaces each 25 ft. diam. and 11 ft. 7 in. high. These furnaces are made of fire-brick lined on the hearth and lower wall sections with carbon blocks. In each furnace there are six carbon electrodes 2 ft. diam., which are powered from transformers at 150 to 170 V. and 5,500 to 7,000 amp. to raise the temperature to 1,300°C. The electrodes are suspended vertically on winches through the furnace roof so that their current can be maintained by vertical adjustment, as shown in Fig. 5. New carbon sections are screwed into the top as soon as the old ones have burned away. Consumption of electric energy is about 6 to 7 kWh./lb. phosphorus produced, and it should be noted that the electric current only provides the high temperature necessary for this endothermic reaction.

The products of the furnace are gas (chiefly carbon monoxide, hydrogen and phosphorus vapour), slag (calcium silicate and ferrophosphorus).

Only 50% of the heat input into the furnaces is used in the reaction; the remainder can be accounted for as follows: 30% in slag, 5% in exit gases, 10% radiation losses and 5% transferred to cooling water. It would seem, therefore, that a more efficient heat utilisation could considerably decrease the electrical consumption.

Since the pressure of gas in the

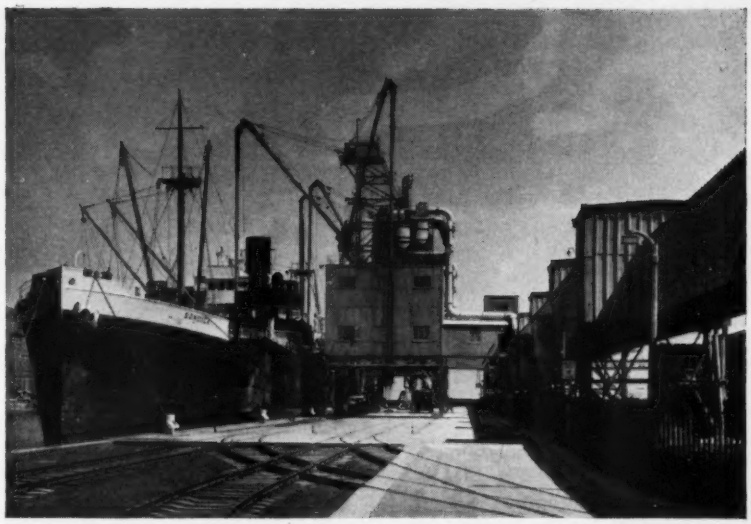


Fig. 2. Ship carrying phosphate rock from Florida standing in the dock at Portishead.

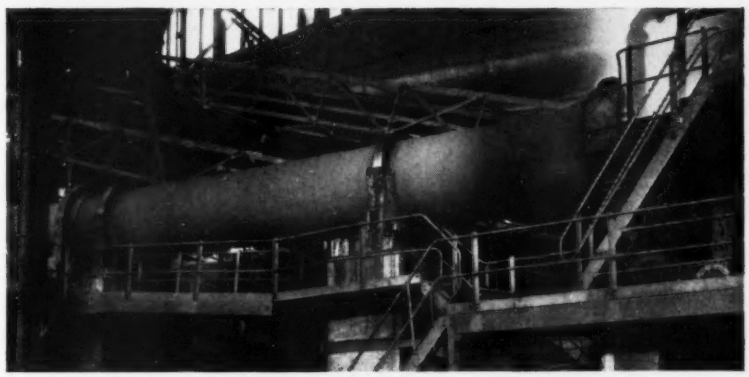


Fig. 3. One of three calciners in the preparation house.

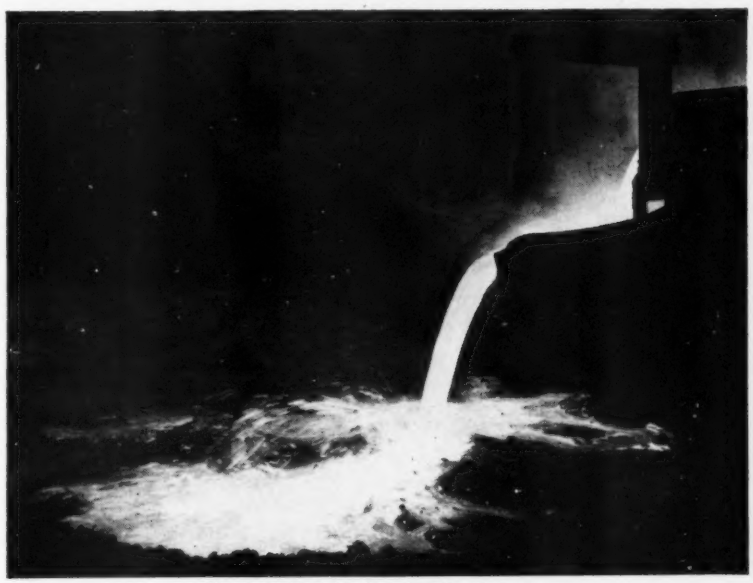


Fig. 4. The slag leaving the furnace.

furnace is only just above atmospheric it is only necessary to seal the gap between the electrodes and the furnace by means of a simple packing. This has the additional advantage of acting as pressure vent to release sudden bursts of flames caused from time to time when the molten mass within the furnace collapses—a frightening experience to the casual spectator.

Condensation

The gas is conducted from the furnaces to two hot condensation towers, set in series, where it is sprayed with hot water. This reduces the gas temperature to about 60 to 70°C. and the phosphorus vapour condenses out as a liquid which is contaminated with solids carried over in the gas stream. For two or three days the liquid is kept under water in settling tanks and during this time the contaminants rise to the top of the phosphorus. The phosphorus is then pumped into storage tanks where it is kept under water until despatch by road or rail. The furnace gas is used in the boiler house for steam raising (ideally this should be adequate to produce all the steam, but variations in the calorific value of the gas have discouraged too much reliance on it as a fuel). The slag when tapped from the furnace, see Fig. 4, is separated into calcium silicate—used for road surfacing, and ferrophosphorus—used in the manufacture of certain steels.

The product is transported mainly in rail tankers which are filled from underground storage tanks. These tankers can hold up to 48,000 lb.

World production of phosphorus in 1958 was about 440,000 tons, of which the U.K. contributed 24,000 tons—the annual production at Portishead is just over 13,000 tons. About 90% of the phosphorus produced in this country is converted to orthophosphoric acid, of which 80% is used in the manufacture of various phosphates. This would indicate that the wet process in which phosphate is decomposed by sulphuric acid to give orthophosphoric acid according to Albrights' original process mentioned above, is both cheaper and simpler by avoiding the conversion of phosphorus to orthophosphoric acid. The next largest uses of phosphorus are in the preparation of phosphorus chlorides, thiophosphoryl chloride, $PSCl_3$, and phosphorus pentasulphide, P_2S_5. A smaller proportion of the output is cast into sticks and wedges for the preparation of phosphor-copper used in the production of phosphor-bronze.

The rapid growth in demand for phosphorus can best be illustrated by the fact that, whereas the quantity of phosphorus used in the match industry has remained constant since 1914, when it represented 75% of the total U.K. production, in 1959 it represented less than 1%.

Fig. 5. The top of one of the six furnaces.

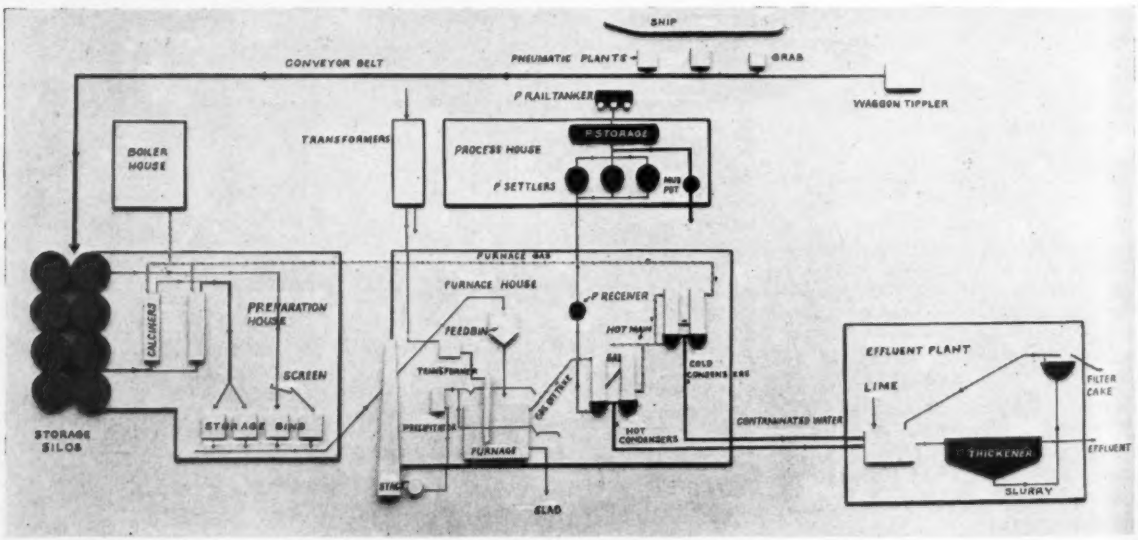


Fig. 6. Flow-sheet of the process.

Chemical Engineering Fundamentals – 2

By K. L. Butcher,* B.Sc., F.R.I.C., M.I.Chem.E.

This is the second article in a series undertaken to re-examine some principles basic to chemical engineering. The ideal gas law, $pV = RT$, is discussed in relation to the principle of conservation of energy. From this can be defined many important concepts used in heat, mass and momentum transfer such as specific heat, thermal conductivity and viscosity. The validity of the ideal gas law is then examined with regard to the influence of inter-molecular forces on this system.

AN understanding of the properties of gases is of importance in chemical engineering calculations. The empirical law

$$pV = RT$$

governing the physical state of 1 gram molecule of an ideal or perfect gas ($R = 0.082$ *l*.atm./°K.) is basic to many approximate calculations. More refined calculations rely upon more complicated empirical formulae relating pressure, volume and temperature. In the light of the interpretation of $pV = RT$ by the simple kinetic theory of gases, the more complicated formulae such as:

$$pV = RT + Bp + Cp^2 \ldots.$$

may be regarded as making allowances for the intermolecular forces of attraction (or repulsion) and the finite volume of the molecules themselves (see Fig. 1). The simple kinetic theory of gases contains the postulates that gas molecules behave like hard, perfectly elastic and perfectly smooth spheres which do not exert any force of repulsion or attraction upon one another. The gas molecules, when contained for a long time in a stationary vessel of constant dimensions, during which no heat flows either to or from the vessel and there are no alterations in the intensity of any external fields (*e.g.* gravitational, electrical or magnetic), come to a state known as equilibrium. It is to this state that the following remarks apply.

The gas molecules are imagined to be in a state of chaotic motion which may be accounted for completely by molecular linear velocities having independent components in three dimensions. If all the gas molecules are of one kind and there are n molecules per unit volume, considering only motion in the positive direction of x, with velocity of magnitude u, then at the wall of the vessel in the yz plane, the molecules colliding with the wall will suffer a reversal of the velocity which assumes a magnitude u in the direction $-x$.

Conservation of energy

This is in conformity with the principle of conservation of energy in which it is stated that a system continues in a state of constant total energy (kinetic plus potential) in the absence of any supply or withdrawal of energy by external agencies. The failure to devise a perpetual motion machine, the definition of force,

$$m\frac{du}{dt} = P$$

and of work

$$\left(\int_{x_1}^{x_2} P dx\right)$$

which is also change in potential energy, underlie the principle of conservation of energy and the definition of kinetic energy, *viz.*:

$$\tfrac{1}{2}mu^2 \text{ or } \int mudu = \int Pudt = \int Pdx.$$

Thus, since no change in potential energy takes place in the idealised reflection of the molecule, the kinetic energy is the same before as after the reflection. The rate of change momentum due to this cause, over unit area of wall, gives the pressure of the gas on the wall. If n is number of molecules of mass m, per unit volume, then $\frac{n}{6}$ molecules on the average are associated with one direction of motion, and thus $v \times 1 \times \frac{n}{6}$ mol./sec. experience the change in momentum $2mu$.

Let n = total number of molecules of mass m, per unit volume. If n_1 mol./vol. have components of velocity (mutually perpendicular), u_1, v_1, w_1, then the pressure on unit area of yz wall can be estimated by considering the average change in x component momentum associated with unit area in unit time.

The n_1 molecules with velocity component u_1 towards yz contribute n_1u_1 impacts per second, each having a momentum change of $2mu_1$. This group therefore contributes a momentum change per second of $2m.n_1u_1^2$. The whole population of molecules will have an effect determined by the appropriate average value of $(n_1u_1^2)$, *i.e.* sum $(n_1u_1^2)$ for all groups moving towards yz divided by $\frac{n}{2}$, since half n will have u_1 positive and half will have u_1 negative, *i.e.*

$$\frac{(n_1u_1^2)}{n/2}$$

This is a definition of a mean of the square of u, *i.e.* $(\overline{u^2})$. Thus $p_{yz} = 2m.(n/2.\overline{u^2}) = nm.\overline{u^2}$. If $\overline{c^2} = \overline{u^2} + \overline{v^2} + \overline{w^2}$ = mean of the square of velocity c having components u, v, w, then, if

$$\overline{u^2} = \overline{v^2} = \overline{w^2} = \tfrac{1}{3}\overline{c^2},$$

$$p_{yz} = \frac{nm\overline{c^2}}{3} = p_{xy} = p_{zx} = p$$

If there are N_{Av} molecules in a gram molecule of gas, then, if this amount has volume V, we have

$$p = \frac{1}{3}\cdot\frac{N_{Av}}{V}\cdot m\overline{c^2} = \frac{1}{3}\cdot\frac{M}{V}\overline{c^2}$$

where M = molecular weight of gas. Hence,

$$pV = \tfrac{1}{3}M\overline{c^2} = RT$$

*Brotherton Senior Lecturer in Chemical Engineering, The Houldsworth School of Applied Science, University of Leeds.

(by idealised experimental gas law). Thus,

$$\tfrac{2}{3}pV = \tfrac{2}{3}RT = \tfrac{1}{2}M\bar{c}^2$$

Heat capacity

Thus, if we regard the gas as possessing energy of one kind, namely kinetic energy, then per gram molecule the kinetic energy or total energy E is

$$\tfrac{3}{2}pV \text{ or } \tfrac{3}{2}RT$$

If the energy is required in cal./g.mol., $R = 1.98$. Thus, at constant volume

$$\left(\frac{dE}{dT}\right)_v = \frac{3}{2}R$$

Since Joule demonstrated the equivalence of work and heat, and mechanics shows the equivalence of kinetic energy and work, we see that the amount of heat required to raise the temperature of 1 g.mol. of gas by 1°C. is $\frac{3}{2}R$. This is in fact the value observed for the specific heat of monatomic gases at constant volume and may be regarded as a contribution of $\frac{1}{2}R$ from each velocity co-ordinate required to define completely the kinetic energy, *viz.*:

$$\tfrac{1}{2}m(u^2 + v^2 + w^2) = \tfrac{1}{2}mc^2/\text{mol.}$$

If the heat be added at constant pressure, then work is done against the atmosphere and the equivalent of this work will therefore be needed in addition. Since p is constant, the work done is $p(V_2 - V_1)$ and if V_2 corresponds to $(T_1 + 1)$ and V_1 corresponds to T_1, we see that

$$p(V_2 - V_1) = R(T_1 + 1) - T_1 = R$$

Thus, $C_p - C_v = R$, and so C_p for a monatomic gas should be

$$\left(\frac{3}{2} + \frac{2}{2}\right)R = \frac{5}{2}R$$

and $$\gamma = \frac{C_p}{C_v} = 1.66.$$

Diatomic gases

Diatomic gases are found not to agree with this theory of specific heats and some have ratios of

$$\frac{C_p}{C_v} = 1.4$$

approximately. This implies $C_v = \frac{5}{2}R$ instead of $\frac{3}{2}R$. By postulating that energy may be held by the molecules not only by rectilinear translation but also by rotations composed of components in the plane containing the molecule (but not about its longitudinal axis) and in a plane at right-angles to this, two additional $\frac{1}{2}R$ terms were introduced. For non-linear tri-

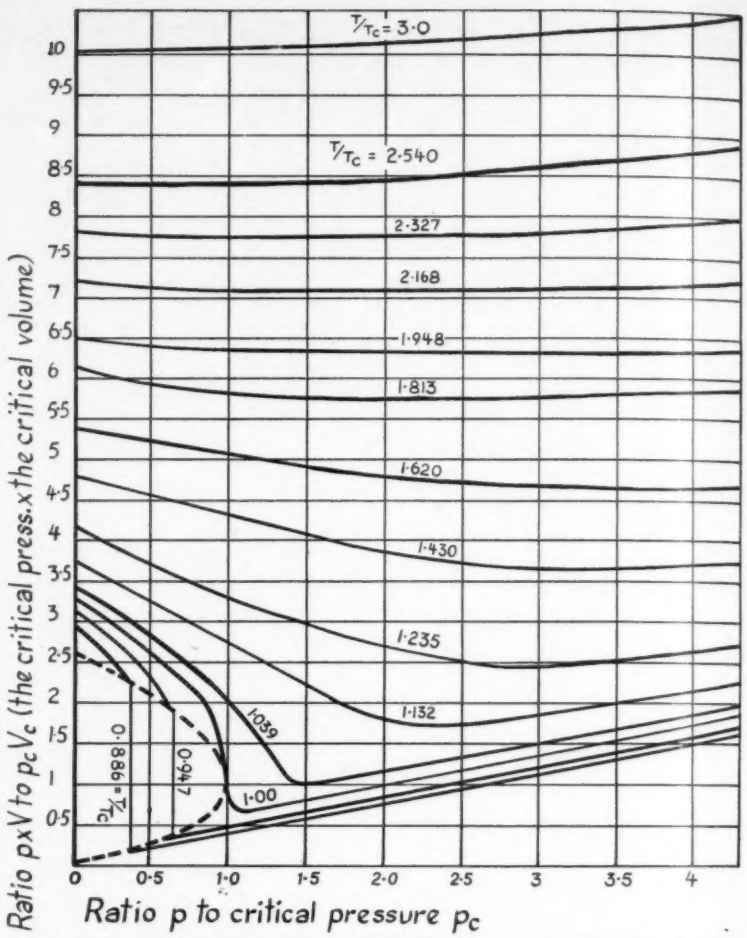


Fig. 1. Pressure volume temperature relationship for any gas of known p_c, T_c and V_c.

atomic molecules a further $\frac{1}{2}R$ term is introduced, because a third axis of rotation is then probable.

However, discrepancies in observed specific heats and theoretical values for polyatomic gases persist especially for higher temperatures than normal and postulates that vibration between atoms in the molecules also store energy, lead to a theoretical value of specific heat which was only realisable as an upper limit if the gas did not decompose. The proper quantitative explanation lies in the conception that energy is not taken up in a continuous fashion by molecules, but in discreet quanta, the distribution of which in the case of vibrational energy in many of the molecules considered is sufficiently temperature-dependent to account for the changing specific heat. Once the fundamental frequency of the vibration is known (from spectroscopic data) it is possible to evaluate the vibrational energy of the gram molecule of gas at a given temperature using formulae derived from quantum and statistical mechanics.

Maxwell-Boltzman distribution

To return to the rectilinear motion, this is distributed among the molecules when they are in equilibrium with one another and their container, in such a way that there are very few with zero motion or with extremely high motion, but increasing numbers as one proceeds to consider intermediate values of motion. The fraction of molecules having a motion between limits c and $c + dc$ (for one T), when plotted against c, gives rise to a 'skew' humped curve (see Fig. 2). The formula for the curve was derived by Maxwell and Boltzman and shows that the fraction d varies as

$$Ae^{\frac{-mc^2}{2RT}}dc$$

where A is a constant. This is important in connection with the velocity

of chemical reactions and may be simply derived from the solution of two simultaneous differential equations, one stating that the rate of change of kinetic energy with time is zero, hence $udu + vdv + wdw = 0$, the other that the fraction of molecules having x velocity component in the range u, $u + du$, is

$$u = \frac{dN}{N} = f(u)du$$

It is assumed that the components are independent of one another.

Thermal conductivity

In addition to enabling one to estimate specific heats of simple gases, the elementary kinetic theory enables one to estimate such properties as thermal conductivity, viscosity and diffusivity of gases. These properties are of importance in transfer processes even though these be mainly convective. The argument put forward is similar for the three cases, and it will be stated for the case of thermal conductivity.

Suppose heat to be flowing under the influence of a small temperature gradient because some of the properties of the gas have to be estimated from 'equilibrium' conditions, and these will not be strictly tenable in the presence of a temperature gradient. It is useful to imagine three layers of gas molecules lying in the yz planes separated by a distance representing that which statistical averaging shows to be the most likely distance a molecule will have travelled since its last collision. The distance is called l. If T be the temperature of the central layer of molecules, then the temperature of the layer nearer $x = 0$ is

$$T - l\left(\frac{dT}{dx}\right)$$

and that of the remaining layer is

$$T + l\left(\frac{dT}{dx}\right)$$

bearing in mind the essential negative nature of $\frac{dT}{dx}$ and taking positive heat flow in the direction of positive increasing x.

Now consider the transport of heat by molecular transportation from one layer to the next. If C_v is the specific heat of the gram molecule and if the member density of molecules is n and uniform, then the net heat transferred per second from left to right across unit yz area is

$$\frac{n}{6} \cdot \frac{C_v}{N_{Av}} \cdot u\left[\left(T - l \cdot \frac{dT}{dx}\right) - \left(T + l \cdot \frac{dT}{dx}\right)\right]$$

The central layer sends out

$$\frac{n}{6} \cdot \frac{C_v}{N_{Av}} \cdot u \cdot T$$

both to left and to right, thus making no net contribution.

Let q = rate of heat flow per unit area along x.

$$q = \frac{-n.C_v}{3\,N_{Av}} \cdot ul\,\frac{dT}{dx}$$

But, by the definition of thermal conductivity k,

$$q = -k.1 \times \frac{dT}{dx} \text{ in this case.}$$

Thus $k = \frac{n}{3} \cdot \frac{C_v}{N_{Av}} \cdot ul$

Now $\frac{n}{N_{Av}}$ = number of g.mol./unit volume; u = average speed of molecules—assumed not to be influenced appreciably by the temperature gradient; and l = mean free path of molecules which may be roughly estimated from the volume a molecule sweeps out in unit time when travelling at mean speed u. If d is the diameter of the molecule this will be the radius of 'collision cylinder' swept out, of which the volume will be $\pi d^2 u$. There will be $n\pi d^2 u$ collisions in unit time. The total mean distance travelled in unit time must be u, so

$$l = \frac{u}{n\pi d^2 u} = \frac{1}{\pi n d^2}$$

Thus $k = \frac{nC_v}{3\,N_{Av}} \cdot u \cdot \frac{1}{\pi n d^2} = \frac{C_v}{N_{Av}} \cdot \frac{u}{\pi d^2}$ which is independent of the number density.

An estimate of u is obtained from

$$\frac{1}{2}Mu^2 = \frac{3}{2}RT \text{ or } u = \sqrt{\frac{3RT}{M}}$$

The value of k =

$$\frac{C_v}{3\,N_{Av}}\left(\sqrt{\frac{3RT}{M}}\right)\frac{1}{\pi d^2}$$

The effective diameter of the molecule needs to be known to evaluate k.

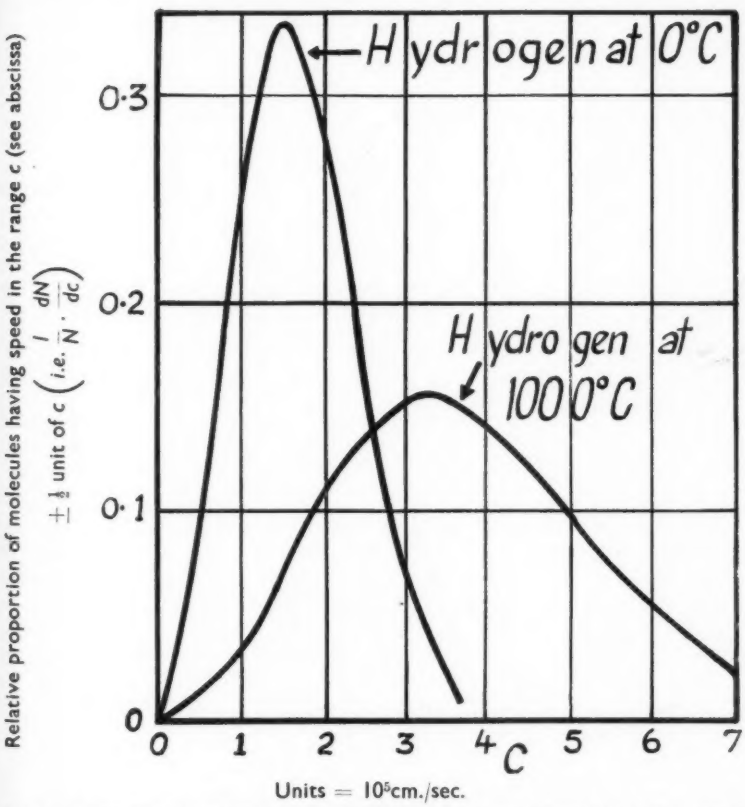

Hydrogen at 0°C
Hydrogen at 1000°C
0·3
0·2
0·1
0
0 1 2 3 4 5 6 7
c
Units = 10⁵cm./sec.
Relative proportion of molecules having speed in the range c (see abscissa) ± ½ unit of c (i.e. l/N · dN/dc)

Fig. 2. Relationship between proportion of molecules with speeds in ($c \pm \frac{1}{2}$) range as a function of c. Total area under each curve for $c = 0$ to $c = \infty$ represents whole of gas, i.e. total proportions = 1.

Since the method of finding average properties is inaccurate, one can only consider the above in a qualitative way. Gases with small molecules will have high thermal conductivities. These conductivities increase as the square root of the absolute temperature and are independent of pressure. The latter, of course, breaks down when the pressure is so low that the distance between the boundary walls in the vessel containing the gas is of the same order as the mean free path l of the gas.

Viscosity

In the case of viscosity, the property transported is momentum. The three layers of molecules now slide parallel to one another in the yz planes, in say the z direction, and have different velocities

$$W = \left(\frac{dz}{dt}\right)$$

of translation relative to the wall of the containing vessel. Three adjacent yz planes distance l apart may be taken as before. The z velocities of these planes will be

$$W - l\left(\frac{dW}{dx}\right) \text{ and } W + l\left(\frac{dW}{dx}\right)$$

The rate of transfer of momentum per unit area must then be the force acting per unit area yz tangentially and in the z direction to maintain the velocity gradient $\frac{dW}{dx}$.

We obtain by the simple argument

$$\text{stress} = \frac{1}{3}nmul\frac{dW}{dx} = \mu ul\frac{dW}{dx}$$

so that the viscosity of a gas $\mu = \frac{1}{3}nmul = \frac{1}{3}nmu\,\frac{1}{\pi nd^2} = \frac{1}{3}\,\frac{m}{\pi d^2}\sqrt{\frac{3RT}{M}}$

independent of density but increasing as $T^{\frac{1}{2}}$.

The self-diffusion coefficient D may similarly be shown to be

$$D_{self} = \frac{1}{3}nul = \frac{1}{3}nu\,\frac{1}{\pi nd^2} =$$

$$\frac{1}{3}\,\frac{M}{\pi d^2} = \frac{1}{3}\sqrt{\frac{3RT}{M}}\,\frac{1}{\pi d^2}$$

The more accurate formulae thermal conductivity k, viscosity μ and self-diffusion constant D are:

$$k = \frac{C_v\sqrt{RT}}{N_{Av}.\pi\sqrt{2}.d^2},\ \mu = \frac{m}{\pi\sqrt{2}.d^2}\sqrt{\frac{RT}{\mu}}$$

where it can be seen that

$$\frac{C_v.\mu}{k} = 1 = \frac{C_p.\mu}{\gamma k}$$

These elementary considerations are of value in the operations concerning heat transfer, fluid flow and mass transfer. At pressures such that the mean free path l is comparable to the width of a tube containing the gas the above ideas break down.

Integration constant

The success of the kinetic theory in the case of gases depended on the fact that a simple law $pV = RT$ applied to them, from which it was possible to evaluate the energy due to the various motions of the gas molecules. Also no intermolecular forces of any consequence are at work in the permanent gases to vitiate the assumptions of the derivations.

Thus the specific heat determinations from theory

$$\left(\frac{dE}{dT}\right)_v = \frac{3}{2}R$$

for monatomic gases agreed with the experimental value at temperatures well above the normal boiling point. However, the integration of such an equation gives, generally,

$$E = \frac{3}{2}RT + E_o$$

where E_o is a constant of integration, and at temperatures not far above the boiling point of the substance, the experimental value of

$$\left(\frac{dE}{dT}\right)_v$$

is itself a function of temperature.

The term E_o may be split up into $E_r + E_v + E_p$, where E_r = molecular rotational energy, E_v = interatomic vibrational energy and E_p is attributable to the mutual potential energy of the molecules which, when the molecules are brought sufficiently close together at constant temperature, part of E_p may be released (given up to the surroundings) first as latent heat of condensation and then as latent heat of fusion, or alternatively just as heat of condensation in the case of direct gas to solid transformations.

It is found experimentally that above a certain temperature T_c, characteristic of each substance, no clear-cut 'condensation' takes place, regardless of the pressure. This temperature is called the critical temperature. Generally speaking, the lower the value of E_p, the lower the value of T_c. Another characteristic temperature of a substance, less independent of pressure, but not very noticeably dependent, is the solid-to-liquid transformation temperature, or melting point. In Fig. 1 is shown the ratio

$$\frac{PV}{P_cV_c}$$

(y axis) as a function of

$$\frac{P}{P_c}$$

(x axis) at various values of T/T_c (numbers on curves). P_c and V_c are the critical pressure.

E_r, the energy contained in the rotation of molecules or parts of molecules, is not generally very temperature sensitive above the normal boiling point of a substance, but E_v may be noticeably temperature dependent, significant at temperatures which in general are higher for high interatomic binding energy, for instance:

H_2	..	800°K. or 527°C.
O_2	..	300°K. or 27°C.
Cl_2	..	100°K. or −173°C.

E_p may itself be regarded as composed of latent heat terms, which are known to diminish to zero as the temperature rises to T_c, and other terms also temperature sensitive, similar in type to those contributing to the 'vibrational' specific heat in the gas molecule but representing inter-molecular vibrations rather than interatomic vibrations, first of the molecule in the solid state, then in an ill-defined way, of the molecules in the liquid state.

There is no generally satisfactory way of evaluating this potential energy term at present. Thus for liquids the specific heat cannot be accounted for quantitatively and empirical expressions for each substance, of the form $C_p = C_{po}(1 + at + bt^2)$, where a and b are numbers characterising the substance (a is usually positive; t = temperature, °C.).

Methods for estimating heat capacities of gases and liquids are given in 'The Properties of Liquids and Gases', by Reid and Sherwood (McGraw-Hill, 1958). Perry gives some data on page 228 of 'Chemical Engineers' Handbook, 1950'.

Thermal conductivity for liquids

The transport properties of liquids (thermal conductivity, etc.) are not predictable to the same extent as those of gases because of the vagueness about the structure of liquids and the intermolecular forces. One proposal for k is superficially analogous in form to that for gases but has no theoretical background and is dimensionally inconsistent in its variables:

$$k = 3.59 \times 10^{-3}\,C_p.\rho\left(\frac{P}{M}\right)^{\frac{1}{3}}$$

(*Concluded on page 323*)

WHAT'S NEW

Plant • Equipment • Materials • Processes

CPE reference numbers are appended to all items appearing in these pages to make it easy for readers to obtain quickly, and free of charge, full details of any equipment, machinery, materials, processes, etc., in which they are interested. Simply fill in the top postcard attached, giving the appropriate reference number(s), and post it.

Industrial fan unit

Thermostatically controlled air heating maintains a uniform overall temperature, cools the air in summer, heats it in winter and keeps up a steady convection flow to avoid patches of stale air.

To meet these requirements General Trade Equipment offer an industrial fan unit. It is designed to eliminate vibration and so avoid short circuiting.

The open-wire spiral-type heating elements operate at black heat under all conditions. The thermostat can be hand set to cut off the current automatically if overheating occurs.

The direct starting motor is fitted with mounted ball bearings. A detachable air filter can also be fitted.

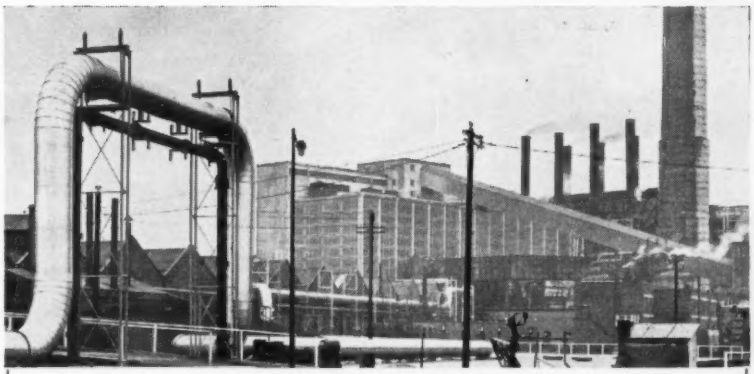
Fans are supplied in seven different sizes, the smallest with a 5-kW. output (17,000 B.Th.U./hr.) and the largest with a 40-kW. output (136,000 B.Th.U./hr.). **CPE 1521**

Flocculation

Aqueous suspensions containing particles with negatively charged surfaces lend themselves to flocculation by cationic chemicals, provided that these are water-soluble and sufficiently low-priced to warrant their use. Such products are the *Arquads* (quaternary ammonium salts) and the *Duomacs* (diamine acetates), offered by Armour Hess Chemicals Ltd.

A typical commercial application for the products is the flocculation and settling of colloidal silica where other conventional flocculants have failed. Mineral silica, clays and iron-ore suspensions are other materials where cationic flocculants are said to give good results.

A particular advantage claimed for the *Arquads*—which are strong algaecides—is that, simultaneously with their use as flocculants, algae growth is effectively controlled. **CPE 1522**



ALUMINIUM CLADDING

Aluminium sheet has been used to clad this steam main at Spondon works of British Celanese Ltd. to give protection from the weather, which is claimed to be lasting and maintenance-free.

The main, a 30-in.-bore steel pipe, was installed to tap steam from a power station and deliver it 2,125 ft. for distribution in the British Celanese works. The system also includes some 1,300 ft. of smaller-bore branch pipes.

All the insulated pipes in the system were protectively finished with Noral *sheet, 0.036 in. thick, supplied in coil form by Northern Aluminium Co. Seven tons of aluminium were needed in all, but on the largest pipe the cladding weighed only a little over 4 lb./ft. and its lightness enabled savings to be made in the supporting structure.*

Lengths of coil were cut to wrap the main circumferentially with a 2-in. overlap joined by Pop *rivets. Adjacent wraps along the length of the pipe also overlapped by 2 in., but a minimum of permanent fastenings was used at this joint to allow for expansion and contraction.* **CPE 1523**

'Natural' synthetic rubber

Goodrich-Gulf Chemicals Inc. are preparing for the commercial production of two new synthetic rubbers which will be marketed in the U.K. by Durham Raw Materials Ltd.

The two new products, *Ameripol* SN rubber (polyisoprene) and *Ameripol* CB rubber (cis-polybutadiene), will be produced interchangeably in the same plant which will have an annual capacity of about 25,000 tons. Testing and evaluation samples are available immediately.

Ameripol SN, the first synthetic rubber with similar molecular structure as natural tree rubber, possesses the same physical properties as tree rubber. It has been produced in five distinct types in tonnage quantities for large-scale testing.

Ameripol CB rubber is claimed to have good characteristics for minimising heat build-up, a prime cause of the destruction of truck tyres. It is said to be a stronger, tougher rubber at high temperatures than any other general-purpose synthetic rubber now available and has higher rebound

characteristics than natural rubber.

The principal ingredient of *Ameripol* CB rubber is butadiene, which is also the main component of SBR general-purpose synthetic rubber. The principal raw material for *Ameripol* SN is isoprene. **CPE 1524**

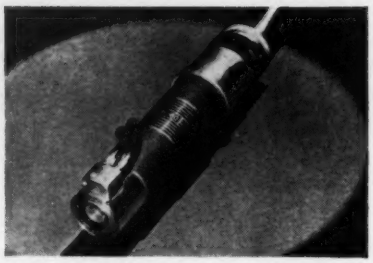
Liquid hydrogen

Pilot quantities of liquid hydrogen are offered in this country on a commercial basis for the first time. The first three deliveries totalling 300 l. were made recently by British Oxygen Research & Development Ltd. to a customer some 70 miles from the producing plant which has recently been installed to enable them to meet demands for small quantities of liquid hydrogen. **CPE 1525**

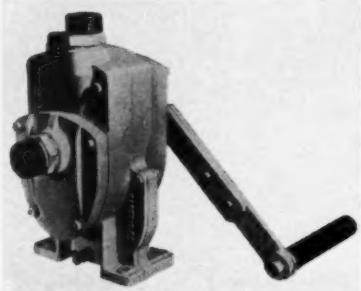
X-ray tubes

New X-ray tubes for X-ray fluorescence analysis in which the characteristic radiation from impurities in the tube anode is reduced to a very low intensity, are made by Machlett Laboratories Inc., is offered by Watson & Sons (Electro-Medical) Ltd.

The spurious characteristic radiation generated in X-ray tubes is one of the factors limiting the sensitivity of the X-ray fluorescence method. The new tubes are claimed to allow an improvement in sensitivity to be obtained, especially for copper and nickel, and the effect of target impurities is said to be now insignificant. The new tubes have beryllium windows and operate up to 50 kV. **CPE 1526**



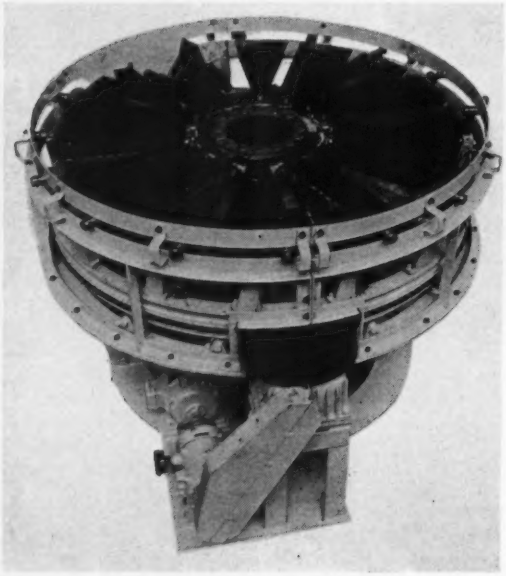
Beryllium window tube for research and fluorescence analysis.



PORTABLE PUMP

A lightweight, portable hand pump which needs no priming and can be put into operation quickly and easily on site or even incorporated into permanent installations is offered by General Trade Equipment Ltd.

The range includes models with a capacity of 150 *gal./min. at* 5 *ft. static delivery head and suction lifts of up to* 32 *ft.* **CPE 1527**



A pilot model of the 'Paxman' rotary table filter.

Flowmeter

The de Havilland *Pottermeter* is a precision electronic flowmeter incorporating an hydraulically positioned bearingless rotor claimed to maintain extreme accuracy over long periods.

It is said to be suitable for use with any liquid irrespective of its lubricating or non-lubricating qualities. It can be fitted into any pipeline size and is no larger than the pipe in which it is installed. It can also be supplied for gas-flow measurement.

The system consists of a sensing element and an indicating or recording unit, which can be placed any distance from the sensing element, to which it is connected by an electric cable.

The flow sensing element is based on the principle of using the kinetic energy of the measured fluid itself to suspend the turbine-type rotor in an axial 'floating' position. Thrust friction is thus eliminated, and the rotor spins freely on plain guides without slippage at a rate governed by and directly proportional to the velocity of the flow through the unit.

Mounted within the body of the standard de Havilland *Pottermeter* rotor is a small permanent magnet, polarised at right-angles to the axis of rotation, and, as the rotor spins, this magnet sets up an alternating current in a coil of wire held within its field. The total number of pulses generated by the sensing element is directly proportional to the total quantity of liquid passing through it.

The frequency of the alternating current produced is directly proportional to the rate of flow of the liquid through the sensing element. **CPE 1528**

Protective coating

An anti-corrosive coating has been developed by Tretol Ltd., which in two coats is claimed to attain a film thickness of 20 mils (equivalent to 15 to 20 coats of normal paint) at a fraction of the cost. The coating is said to have resistance to all forms of attack by acids, alkalis, sea-water, oil, sewage and brine to an extent not previously possible by brushed application.

Adhesion is obtained without priming on metals and concrete. It is recommended for protecting plant, pipework, tanks, coke-oven equipment and dock and harbour installations. **CPE 1529**

Rubber processing equipment

An agreement has taken place between Baker Perkins Ltd. and the German firms of Leonh. Herbert and Paul Troester, which allows the British firm to manufacture and sell

a complete range of rubber processing and moulding machinery previously made in Germany.

Leonh. Herbert are designers of bladder-type tyre curing presses both for new and retread work. A recent addition to their range is a series of mechanically operated presses for general moulding.

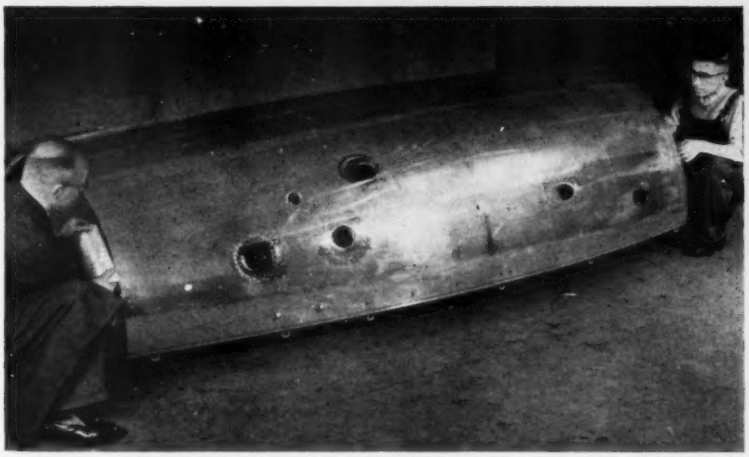
Paul Troester supply standard rubber processing equipment—including mills, calenders and extruders. Their complete range has been redesigned recently to meet the present-day trend towards higher output. **CPE 1530**

Table filter

A continuous rotary table filter suitable for filtrates as widely differed as caustic lime slurry, brewers' mash or oilseed pulp is offered by Davey Paxman & Co. Ltd. It is available in a variety of construction materials, offers several stages of cake washing during one cycle of operation and can be fitted with a vapour-tight hood for use with volatile liquids.

The filter operates on the suction principle. It is intended for use with the quick-settling types of slurries which in the past have presented cake-washing problems and at the same time it is claimed to overcome many of the difficulties connected with the abrasive nature of some of the materials processed.

The final product is said to be clean cake containing as little as 6% moisture from liquids carrying coarse solids, although the amount of residual water will depend on the nature of the raw material. **CPE 1531**



Protecting the highly finished surface of the Boeing engine cowlings at Rolls-Royce aero engine division using 9-in. 'Sellotape' vinyl protection tape. The previous method of protection was by spraying.

Protection tape

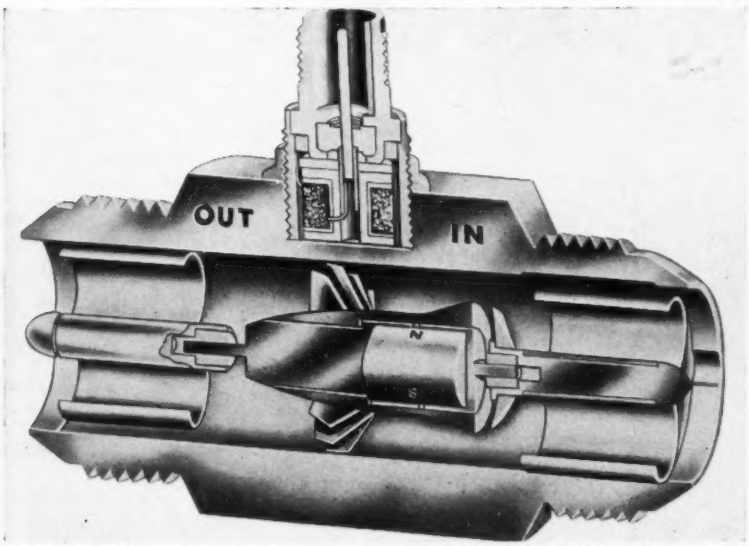
A new vinyl protection tape has been added to the Gordon & Gotch Ltd. *Sellotape* range. It can be applied to a wide range of surfaces including polished steel, polished aluminium, anodised aluminium, mild steel, lacquered or painted metals, decorative and industrial plastic laminates, porcelain, glass and some polished wood surfaces.

This transparent, unplasticised vinyl film, claimed to give a good measure of pliability, is coated with a special low-tack adhesive designed to give both ease of removal from all surfaces and long application life.

It is not intended to give absolute protection against severe wear and tear and abrasion, but to provide a barrier against scratches, disfiguring marks and scuffing which would make many polished or decorative surfaces unsuitable without expensive refinishing.

The film is claimed to be fully water and moisture vapour proof. It also is said to exhibit a high degree of resistance to attack by chemicals, thus offering an all-round protection hitherto impossible with conventional tapes.

To limit the possibility of rusting on steel surfaces a vapour-phase inhibitor has been incorporated in the adhesive. **CPE 1532**



A cut-away 'Pottermeter' showing the magnet in the rotor and the coil mounted outside the housing.

Radiation testing

An environmental chamber that will test products and materials while undergoing radiation exposure, developed by Cincinnati Sub-Zero Products (U.S.A.), is offered by Farnell Instruments Ltd.

The 3-cu.ft. test chamber has an adjustable temperature range from +180°F. to –80°F. Altitude control from sea-level to 100,000 ft. can be accomplished in 15 min. A steam vapour generator provides up to 100% relative humidity in the chamber. The water supply reservoir is mounted directly on the unit so that, except for the electrical service, it is completely portable on four 6-in. casters.

Radiation in the chamber is accomplished by a 1-in.-diam. non-metallic tube that penetrates through the chamber and extends 6 in. on each side. A capsule of radioactive material is inserted in the tube for the test.

The chamber can be used with or without the capsule in the tube.

Other special accessories of the unit include programme controllers for wet and dry bulb temperatures and absolute pressure. **CPE 1533**

Polybutadiene rubber

The old methods of activating polymerisation of butadiene with sodium or potassium yielded rubbers which were inferior to the SBR types. The application of stereospecific polymerisation with Ziegler organometallic catalysts, consisting of aluminium alkyls with titanium halides, produces a polymer of high cis-1, 4-structure. This product approaches properties similar to natural rubber with an increasing cis-1, 4-portion of the polymer. The tensile properties are lower than those of natural rubber, yet hysteresis and elasticity qualities are excellent and oxidation resistance is good as is shown by the physical properties:

Specific gravity	0.94
Tensile strength, vulcanised (kg./sq.cm.):	
Unreinforced	20—70
Reinforced	110—200
Elongation (%)	300—500
Wear resistance	Very good
Tear resistance	Fair
Elasticity	Outstanding
Crack resistance	Good
Maximum service temperature range	−90°C.
Light resistance	Poor
Oxidation resistance	Very good
Oil resistance	Very poor
Swelling resistance in contact with:	
(*a*) Aliphatic	Very poor
(*b*) Aromatic	
(*c*) Halogenated solvents	

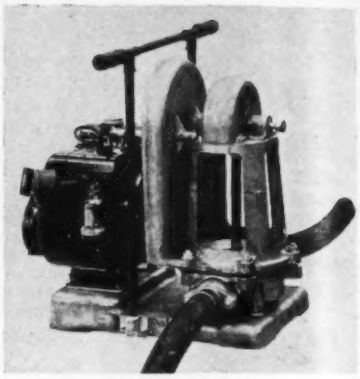
The makers, Chemische Werke Hüls, state that the product can replace natural rubber to a large extent and therefore rivals cis-1, 4-polyisoprene in future importance. **CPE 1534**

Fill in and post the reply-paid card for details of any items in these pages, making sure to quote the correct 'CPE' reference number.

Diaphragm pump

A 2-in. diaphragm pump made of aluminium is offered by Lee, Howl & Co. Ltd. The pump can be easily carried by two men (total pump weight 124 lb.).

It is recommended for liquids containing solids and also oil and chemical fluids of many kinds.



Diaphragm pump.

The diaphragm is of the pinched type, which is said to eliminate wear on the diaphragm and stones becoming trapped beneath the outer edge. The 'straight through' design also discourages stones from settling in the pump body.

Both the flap valve and the diaphragm are made from neoprene, and other parts can be specially lined, so that a wide range of chemicals can be handled.

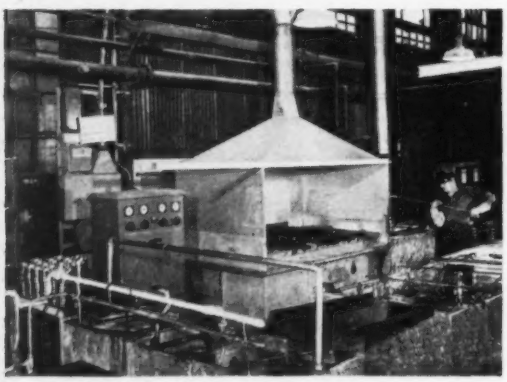
The pump has been designed to run at 90 r.p.m., this low speed being obtained by using a 4-to-1 chain reduction drive. The steel countershaft of the pump runs in oil-retaining bearings and carries cast-iron eccentric with gun-metal straps and a steel connecting rod. The pump has a safety device in that it incorporates a shear pin in the driven chain wheel boss. **CPE 1536**

Store, weigh and feed machine

Wm. Gardner & Sons (Gloucester) Ltd. have designed a machine to store, weigh and feed, at a predetermined rate, materials which are normally difficult to handle satisfactorily such as wood shavings, soft brown sugar and damp granulated products.

It is based on a bunker fitted with an internal slow-running agitator which keeps the materials 'live' within the bunker and prevents any tendency to bridge. A small volume of the 'live' material is delivered through an opening in the side of the bunker and the same total volume is always present within a container at the side of the bunker. By this means, the density of the material available is not likely to vary due to a variation in the amount present in the main bunker.

The degree of accuracy possible is dependent on the type of material, but with such widely differing materials as wood chips and castor sugar, accuracy better than $\pm 1\%$ by weight is said to have been achieved. **CPE 1535**



A 'Cassel Ajax' electric furnace with pot 58 in. × 20 in. × 20 in. deep, used by Hoffman Manufacturing Co. Ltd. for austenising large and special sizes of ball and roller bearing races before quenching in a 'Cassel' 72 in. × 24 in. × 24 in. deep furnace. Standard size of race are treated in three continuous automatic furnaces.

Furnaces

Five new types of electrically heated furnaces have been added to I.C.I.'s range of oil- and gas-fired *Cassel* furnaces. Made by the company's general chemicals division and sold under exclusive licence from the Ajax Electric Co., U.S.A., they are available in various sizes.

Three of the new types are electrode-heated furnaces with rectangular pots. The salt in the pots is heated by current passing between electrodes immersed in the salt, and the life of the pot is therefore far longer than that of pots heated externally by gas or oil. Continuous operation is recommended.

The two remaining *Cassel Ajax* furnaces, for lower-temperature working, are fitted with immersion heaters. Here the heat is produced in resistance elements immersed in the salt and suitably protected. So, in these furnaces also, the pot does not have to transmit heat, and should last several years. **CPE 1537**

Personal Paragraphs

★ **Dr. A. W. Baldwin,** an associate research manager of the dyestuffs division of I.C.I. Ltd., retired at the end of May after 38 years with the company and its predecessors. His name has been associated with many new long-chain products including the *Lissapols* and *Velan.*

★ **Mr. D. E. Rooke** has been appointed as development engineer to take charge of the new development and planning section which the Gas Council is establishing. Since January of last year he has been development engineer of the South Eastern Gas Board. He was one of the technical team which sailed in the *Methane Pioneer* on the voyage which brought to Britain the first load of liquid natural gas carried across the ocean.

★ **Mr. J. J. Garland** has been appointed assistant manager of acid sulphur products of Olin Mathieson Chemical Corporation. He was sales representative for industrial chemicals in the corporation's Philadelphia office.

★ **Mr. J. Martin** has been appointed to the board of Mather & Platt Ltd. as head of the pump department. For the last ten years he has been the managing director of Mather & Platt (S.A.) (Pty.) Ltd. in Johannesburg and for 16 years prior to 1949 he was on the staff of the London office of Mather & Platt Ltd.

★ **Mr. J. Howlett,** general manager of the Hull site of the Distillers Co. Ltd., has been appointed a divisional director of the company. He has been with the company for 36 years beginning with analytical work at the research station at Hammersmith.

★ **Mr. R. H. Dodd,** managing director of Chemical Construction (G.B.) Ltd., has been elected a member of the council of the British Chemical Plant Manufacturers Association.

★ **Mr. J. B. Adams,** director of the proton synchrotron division, was appointed, after an emergency meeting, interim director-general of the European Organisation for Nuclear Research (C.E.R.N.). This appointment became necessary as the result of the death in an aeroplane accident of Prof. C. J. Bakker.

★ The death has occurred of **Sir Oswald A. Scott,** K.C.M.G., D.S.O., chairman of directors of Detel Products Ltd.

★ **Mr. G. H. Doust,** deputy general manager since 1958, has been appointed general manager of Plessey International Ltd. Previously he was with the company's chemical and metallurgical division where he controlled the commercial and sales department.

★ **Mr. F. Drake Parker** has been appointed managing director and **Mr. D. H. Carter** director and general manager of McKee Head Wrightson Ltd. Mr. Parker was formerly vice-president of the American company Arthur G. McKee, and Mr. Carter was director and general manager of Head Wrightson Processes Ltd.

Mr. J. J. Garland.

Mr. G. H. Doust.

Mr. J. Martin.

Mr. D. H. Carter.

Mr. M. L. Jofeh.

Mr. F. D. Parker.

★ **Mr. M. L. Jofeh,** O.B.E., manager of the industrial division, has been elected as an additional director of Sperry Gyroscope Co. Ltd. He joined the company in 1947 as an equipment engineer, subsequently heading the engineering unit at the company's Stonehouse factory for a number of years.

★ **M. Georges Claude** died in Paris recently at the age of 89. His observations (with A. Hess) of the high solubility of acetylene in acetone led to the present method of storing and supplying acetylene. In 1903 he introduced a modification of Linde's process for separating oxygen and nitrogen which enabled practically pure gases to be obtained. He also invented an apparatus for the manufacture of synthetic ammonia which was an improvement on the Haber process.

★ **Mr. B. S. Smith** has been appointed as head of the isotope research division of A.E.R.E. Harwell. He was formerly head of health physics division of A.E.R.E. Harwell and his successor in that post is **Mr. N. G. Stewart,** at present at the Authority's Dounreay Experimental Reactor Establishment.

★ **Mr. H. Hadden** has been appointed to the board of directors of Chemstrand Ltd. His appointment as vice-president and general manager, overseas division of Chemstrand Corporation, was announced recently. He is also secretary to the corporation.

★ **Mr. S. H. Bailey,** publicity manager of Tufnol Ltd. since 1934, retired at the end of May after 46 years with the Ellison companies. He is succeeded by **Mr. D. I. Herbert.**

★ At the annual general meeting of the Fertiliser Society, **Dr. H. L. Richardson** was elected president and **Dr. G. W. Cooke** vice-president. **Dr. J. H. Hudson, Mr. A. H. Kaye** and **Mr. J. H. H. Peak** were elected to fill vacancies on council.

★ **Dr. G. B. Cook** has been appointed as chief chemist of the International Atomic Energy Agency. He has been closely associated with chemistry aspects of atomic energy developments ever since its initial stages in England 20 years ago. He will be mainly occupied with work at the Agency's main laboratory at Seibersdorf near Vienna.

★ **Dr. R. O. Roblin** has been elected as president and director of the Cyanamid European Research Institute in Geneva. He is the co-discoverer of the widely used sulphur drugs sulphadiazine and sulphamerazine and vice-president for research and development of American Cyanamid Co. He takes over from **Dr. R. C. Swain** who was recently appointed director general of Cyanamid International. **Mr. D. M. Scarlera,** assistant to the general manager of Cyanamid's central research division, has been elected vice-president of the Institute.

NUCLEAR NOTES

Control and instrumentation

The fifth U.K.A.E.A. reactor school course on the control and instrumentation of reactors will be held from September 12 to 23, and will be open to British and overseas students. It will take place at Durley Hall, Bournemouth.

The course is primarily intended for those who have a direct interest in the control and instrumentation of nuclear reactors, and it will be assumed that participants have some knowledge of the basic principles of these subjects.

The topics to be treated will include: elementary reactor physics; revision of elementary reactor kinetics, control and instrumentation; automatic control of reactors and nuclear power plants; application of computors; reactor transfer functions and function analysers; neutron counting equipment; neutron flux detectors; neutron flux scanning; choice and siting of flux detectors; reactor safety circuits; health physics instruments; flow measurements; reactor temperature measurements; burst slug detection; design of control mechanisms; spatial instabilities in reactors; data reduction problems in large reactors.

In addition to the lectures there will be visits to zero energy and high-flux research reactors and other relevant parts of the Harwell establishment. There will also be a visit to the Atomic Energy Establishment, Winfrith.

DOSIMETRY PROJECT

Dosimetry experiments have been carried out at the Boris Kidrich Institute at Vinca, near Belgrade, under the direction of the International Atomic Energy Agency. Contributions to this international project were made by nuclear energy authorities in Yugoslavia, France, U.S. and the U.K.

The dosimetry project is designed to obtain further data on the effect of radiation on man and, in particular, to assess more precisely the radiation doses received by some persons during a brief uncontrolled run of the Vinca zero power reactor on October 15, 1958. The photograph shows one of four phantoms used in the experiment.

Technical assistance mission

The board of governors of the International Atomic Energy Agency approved the director general's proposal that a preliminary technical assistance mission be sent to some countries in the Mediterranean/African area.

The countries which have requested a visit of the mission are Greece, Morocco, Tunisia, Sudan, the Ivory Coast and the Federation of Mali (the latter two requests submitted by the French Government on behalf of these two republics of the French community).

The mission will collect information and advise on atomic energy programmes in the countries visited and will help in formulating requests for assistance which the International Agency is in a position to render.

The team will include experts in the agricultural and medical applications of radioisotopes, nuclear research and power, training of scientists and technicians, prospecting and mining of nuclear raw materials. It is expected that the mission will leave Vienna at the end of April and stay for approximately one week in each country.

American-German agreement

The Gutehoffnungshutte Sterkrade A.G. of Germany has entered into a long-term licence agreement to manufacture General Dynamics Corporation's major advanced nuclear reactor systems for research and for commercial power generation.

The agreement gives the German firm exclusive manufacturing and sales rights in the Federal Republic.

Austrian reactor

M. & C. Nuclear Inc., a subsidiary of Texas Instruments Inc., will fabricate 36 nuclear fuel elements for ASTRA (adapted swimming pool tank reactor, Austria) under a contract from the Austrian Atomic Energy Society.

The reactor was designed and will be built by A.M.F. Atomics, a division of American Machine & Foundry Co., for the Austrian National Research Centre in Seibersdorf. It will be used jointly by the Austrian Atomic Energy Society and the I.A.E.A.

THE SAVANNAH

The picture shows the assembly of the core of the nuclear merchant ship Savannah *which has achieved a sustained chain reaction at the Babcock & Wilcox atomic energy division laboratory at Lynchburg, U.S.A.*

The core's 32 fuel elements, which contain uranium pellets, are enclosed in 'cans' visible along the circumference of the inner tank. Suspended above them are cruciform-shaped control rods, which are used to regulate the reactivity and to compensate for fuel consumption. To start up the reactor, the core was flooded with purified water and the control rods were raised by remote control. The completed test confirmed nuclear design and engineering calculations, and provided first-hand experience for the safe installation of the core aboard the Savannah. *The core contains sufficient fuel to propel the ship more than* 350,000 *miles without refuelling.*

Display in Egypt

The first operating reactor in the U.A.R. was furnished by Advanced Technology Laboratories, a division of American-Standard. It is a portable research and teaching reactor, used for an operating reactor display in Cairo in May by the U.S. Atomic Energy Commission to demonstrate the role of reactors in academic, industrial, medical and agricultural research. It was open for viewing when not in operation, to show essential components and display U.S. industrial production techniques in the fabrication of reactor systems.

Lucas Heights

Australia's second nuclear reactor will be built for the Australian Atomic Energy Commission at Lucas Heights Atomic Research Station near Sydney.

This will be constructed by Advanced Technology Laboratories of California.

It will be smaller and simpler than the first reactor, HIFAR, at Lucas Heights.

It will produce only 1,000th of the heat generated in HIFAR, and will be used for tests and measurements where intense radiation is not necessary.

The new reactor will be available to all Australian universities for their own research and training programmes.

Fuel materials

A new international company was formed recently to produce nuclear fuel materials.

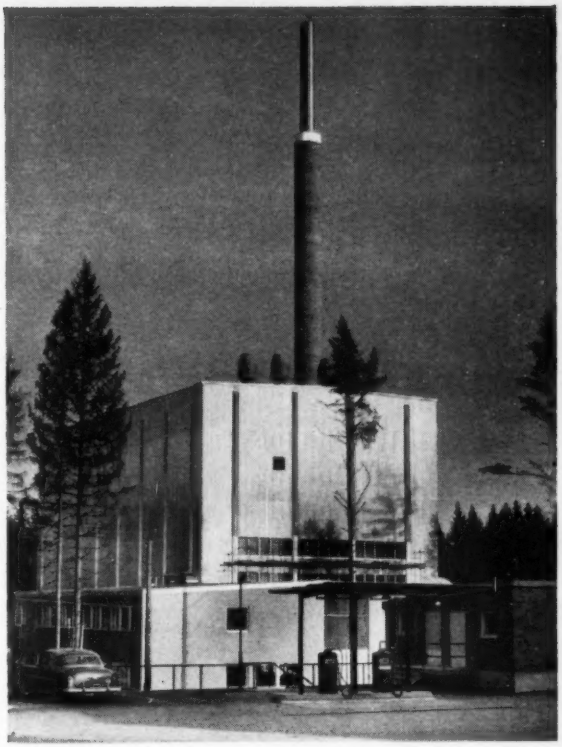
The firm is being created by Societa Montecatini of Italy, Anglo-American Corporation of South Africa and two U.S. concerns, Engelhard Industries Inc. and Mallinckrodt Chemical Works.

U.S.-U.S.S.R. co-operation

The U.S. and U.S.S.R. began exchange visits of nuclear scientists in May. Five American scientists visited the U.S.S.R. high-energy physics establishments and five Soviet experts visited controlled thermo-nuclear research installations in the U.S. These visits implement some of the provisions of the memorandum on co-operation for the peaceful uses of atomic energy signed last November by the chairman of the U.S. Atomic Energy Commission and Prof. V. S. Emelyanov, the head of the U.S.S.R. main administration for utilisation of atomic energy.

The memorandum provides for the exchange of visits and information between scientists of the two countries in the fields of controlled thermo-nuclear research, power reactors development, neutron structure, neutron physics and high-energy physics, the exchange of unclassified technical information and discussions concerning the feasibility of engaging in joint projects in various unclassified areas.

SWEDISH REACTOR



The housing of Sweden's and Europe's largest research reactor which became operative in May. Designed for a rating of 30,000 kW., the unit is installed at AB Atomenergi's research station at Studsvik, on the Baltic Coast, south of Stockholm.

Reactor patented

The first United States patent issued to a private company for an entire atomic reactor system has been granted to North American Aviation Inc. for a reactor developed by its Atomics International division.

The patent was issued in the name of Mr. J. W. Flora, group leader of reactor kinetics for Atomics International, and is for the laboratory reactor designed by him and built by the company.

The 'solution-type' research and training reactor is designed for educational, medical, industrial and biological research. It also produces radioactive isotopes for associated nuclear experiments.

It is 8 ft. high and 8 ft. diam., is easily transported and can be installed without adding special facilities. It is fuelled by enriched uranium in solution.

Other laboratory reactors built by the company are already in operation.

Winfrith ceremony

A ceremony took place recently to mark the start of the construction of the experimental reactor which is being built at the A.E.R.E., Winfrith, under the O.E.E.C. reactor project DRAGON. A steel cylinder containing plans of the reactor, a copy of the project agreement and coins from the signatory countries was laid in the concrete foundation slab of the reactor building by Dr. S. Eklund of Sweden, chairman of the DRAGON board of management.

A 20-Mw. high-temperature gas-cooled reactor is to be built on this site by an international team drawn from Britain and 11 other European countries, which will be ready for operation in 1963. This joint research and development project will help to lay the foundation for the commercial application of this type of reactor for the generation of electric power at a cost competitive with fossil fuels.

Ghana in I.A.E.A.

The application of the Government of Ghana for membership of the International Atomic Energy Agency was unanimously recommended for general conference approval by the Agency's board of governors.

New Books

Power Cost Normalization Studies Civilian Power Reactor Programme, 1959. By Sargent and Lundy. Published by United States Atomic Energy Commission, January 1960. $2.50 net.

'It's an ill wind', as the saying goes, and the development of nuclear power has certainly derived good from the growth of atomic weapons programmes, not the least in the U.S.A. The urgent search for uranium during the past 15 years has at last produced plentiful and cheap supplies of basic nuclear fuel; while the construction of great diffusion plants has brought the price of separated U^{235} down to a level at which a wide range of reactor designs can now be contemplated.

The civil user of nuclear power has to dissociate himself from the 'cost-plus' mentality of defence programmes, and to face up to true commercial conditions, if his product is to be adopted on a wide scale. With current surpluses of coal and oil, the achievement of competitive power is no easy task, and it need come as no surprise that the cheapest source of nuclear power that could be imagined in the U.S.A. last year, for immediate application, was twice as dear as conventional power. In this country last year the margin was seen as only about 30%, both because of the higher cost of coal and the lower nuclear fuel costs associated with natural uranium and bulk processing. Further developments are expected to make nuclear generation for base-load purposes cheaper than conventional power in Britain by about 1970.

Perhaps the most striking feature of nuclear power costs at the moment is the rapid improvement shown by successive installations based on the same reactor principle. These improvements in cost have resulted substantially from engineering advances within the same overall technology. It is perhaps a weakness of the volume under review that no account is taken of this sort of trend. Although eight different reactor concepts are described, the figures all relate to the state of the industry in the year 1959, so that there is no indication of how much further scope each particular system might be thought to have in hand.

It is almost self-evident that a study carried out at this early stage of the art must contain many uncertainties at points where long periods of time are involved. For example, the differences in fuel burn-up which are attributed to the various reactors already seem rather arbitrary. As might be expected, the hydrogen-moderated systems which have received the most development in America come out cheapest—the pressurised-water reactor first, followed by the boiling-water reactor, the organic-liquid cooled reactor, and (rather improbably) the little favoured homogeneous aqueous reactor. The gas-cooled graphite-moderated reactor, which has received so much attention in this country, was regarded as one of the most costly in America, while the fast reactor (which is currently thought to have great long-term potential as a plutonium-fuelled breeder) was rated (on 1959 technical knowledge, including a zirconium-clad metallic uranium/molybdenum fuel) the dearest of all.

There are other features of this study which throw light on the economic differences between the U.S. and this country. The U.S.A.E.C. use higher interest charges on plant and buildings, but use lower interest charges on fissile material, a load factor of 80% (compared with 75% assumed here) and a longer reactor life time of 30 years (compared with 20 years assumed here). For these reasons, and because of variations in costs of labour and materials, it is not easy to take a simple view on the apparent differences between the reactor development programmes of the two countries.

The volume includes a full statement on U.S.A.E.C. prices and credits for nuclear materials, and associated charges. Considerable space is given to assessing the cost differences between medium-size and large reactor installations, *i.e.* between 70 MW(E) and 300 MW(E). It appears that power costs, at least on the American assumptions, rise by about 50% in going to the smaller throughputs, although the organic-liquid cooled reactor suffers less in this respect than the others.

The U.S.A.E.C. are to be congratulated on having attempted a normalisation study, eliminating the numerous individual differences which spring from design teams working to different standards, and with varying degrees of optimism. The results are sufficiently interesting to justify standardising the 'ground rules' and we may look forward to further studies which assess the degree of development attributed to each system on the same basis. The long-term prospects of nuclear power remain very good, and we can welcome this impressive power cost study for the help it gives in deciding the next steps to be taken.

J. L. GILLAMS,

(Mr. Gillams is attached to the Economics and Programming Branch of the U.K.A.E.A. and represents the U.K. at discussions on cost of nuclear power held under the auspices of I.A.E.A.)

Translation from German for Chemists. By H. H. Neville and W. E. Shute. Blackie & Son Ltd., 1959. Pp. 139. 15s. net.

The notorious lack of linguistic ability amongst British scientists is well known and beginning to worry the scientific authorities. During his university training every science student is obliged to acquaint himself with a working technical knowledge of at least two European languages. This may satisfy the consciences of deans of science faculties, but the net gain by most scientists after graduation is very small. What is this due to? Most people will readily ascribe it to the lack of linguistic ability of the Anglo-Saxon people, who have no need to learn foreign languages as long as foreigners prefer to speak English. In contrast to this the Dutch and Scandinavians must of necessity speak two or three foreign languages, which is constantly impressed on English scientists whenever they attend international conferences. Yet it must be remembered that the German language which is spoken and understood throughout Germany, Austria, Switzerland and Central Europe has a vast scientific tradition and no scientist worth his salt can afford to ignore the numerous German publications—or just to rely on their abstracted translations.

The book reviewed here tries to bridge this gap by presenting a short course in basic German for the chemist. The authors have clearly stated that they assume no previous knowledge of German, and have suggested that the reader who works through the book systematically should be able to translate texts set in examinations for science degrees as well as most scientific journals. The approach represents a fusion of methods developed independently by the authors, who are a chemist and German scholar

respectively. The book is divided into 18 chapters, each chapter dealing with a different grammatical case, and after every few chapters some translation practice is interspersed. Altogether it is packed with useful grammatical matter and undoubtedly, if studied consistently, would yield a smattering of technical German. The only doubt is, of course, whether it will ever be read systematically. Unless this is the case the reader might well become afflicted with the disease known as 'a little learning is a dangerous thing'.

I.L.H.

Principles of the Extraction of Metals. By D. J. G. Ives. Royal Institute of Chemistry Monographs for Teachers, No. 3, 1960. Pp. 57. 6s. net.

The monograph is divided into four sections. The first, 'Thermodynamic Background', commences with a discussion of the second law followed by entropy, Gibbs' free energy and chemical potential. This section is not intended as a first introduction to thermodynamics, and readers will interpret it in the light of their knowledge of the subject. In section two, 'Thermodynamics of Pyrometallurgical Processes', the fundamental principles used in the production of metals are discussed with the aid of ΔG°-temperature diagrams.

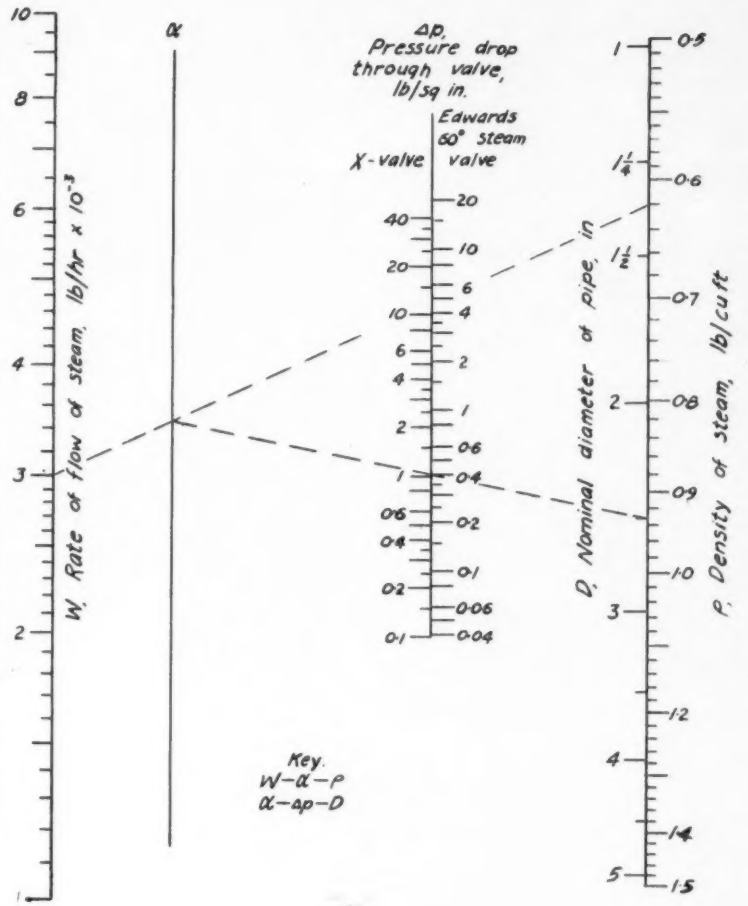
Section three, 'The Choice of Extraction Methods', gives a very brief description of the parting of the elements, then using a modified form of the periodic table it is shown how the metals may be classified into five groups for the choice of extraction methods. The ion-exchange technique for the recovery of uranium is mentioned as well as some special processes which are peculiar to certain metals. The last section, 'Some Typical Processes', describes the operations of roasting and smelting. The iron blast furnace and the production of copper are described as examples of pyrometallurgical processes. A materials balance is given for a blast furnace, but this might have been shown in a diagrammatic form rather than as a table. The factors to be considered in the preparation of a heat balance are also listed. The production of gold is used as an illustration of a hydrometallurgical process.

This publication contains a surprising amount of information. It is well written and can be recommended to all who lecture on this subject.

E. W. JACKSON

Pressure Drop for Steam through Valves

By Prof. D. S. Davis*



When steam flows through valves, the pressure drop can be calculated by means of the equation[2]

$$\Delta p = \frac{B W^2}{D^4 \rho}$$

where Δp = pressure drop, p.s.i.
B = 2.80×10^{-6} for X-valves and 1.075×10^{-6} for Edwards 60° steam valves
W = rate of flow of steam through valve, lb./hr.
D = nominal diam. of pipe, in.
and ρ = density of the steam, lb./cu.ft.

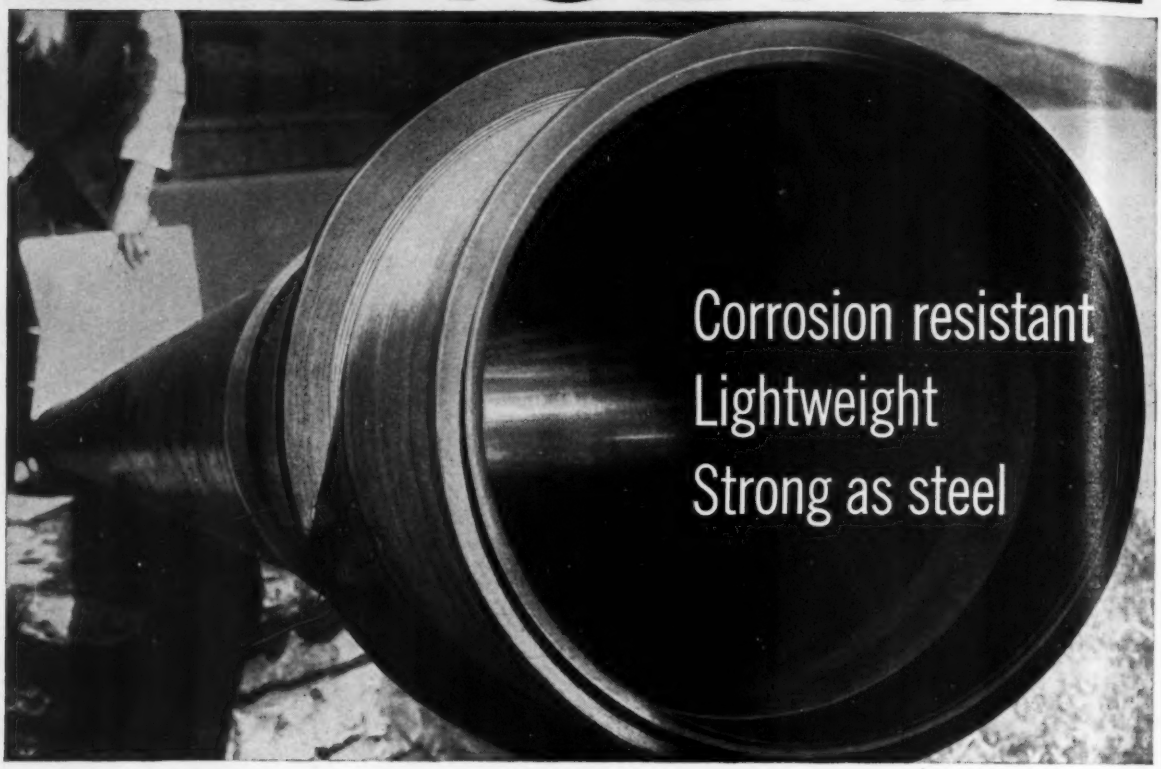
The use of the chart, which was constructed in accordance with methods described previously,[1] is illustrated as follows: What is the pressure drop when steam (density, 0.62 lb./cu.ft.) flows through an X-valve and an Edwards 60° valve in a 2½-in. pipe at 3,000 lb./hr.? Follow the key; connect 3,000 on the W-scale and 0.62 on the ρ-scale with a straight line and note the intersection with the α-axis. Connect this point and 2½ on the D-scale with a straight line and note the intersection with the Δp-scale at 1.04 p.s.i. for an X-valve and at 0.40 p.s.i. for an Edwards 60° valve.

REFERENCES

[1] D. S. Davis, 'Nomography and Empirical Equations', Chap. 6. Reinhold Publishing Corporation, New York, 1955.
[2] *Nat. Pet. News*, 1945, **37** (23), Sec. 2, R 421.

* *University of Alabama.*

BRISTOL PIPE



Bristol Plastics have developed a range of high-precision pipes which has a wide variety of heavy-duty applications. Wherever problems of corrosion or weight interfere with efficient pumping or installation, Bristol pipes offer many big advantages over conventional steel piping.

Bristol Pipe Construction

Bristol pipes are made up of immensely strong helical windings of glass cords embedded in epoxide resin and lined with an abrasion-resistant synthetic fibre matrix which has a glass-smooth surface. Bristol pipes can be manufactured at present in any size from a 6-inch to a 15¼-inch bore, and production techniques are being developed to increase the available bore diameters to 3 feet.

Bristol Pipe Properties

Bristol pipes are highly resistant to corrosion. They need no protective sheathing and can handle liquids, including dilute acids and other corrosive chemicals at high pressures up to 600 psi and above, and up to 140° C.

Bristol Pipe patented couplings.

Bristol pipes are *only one quarter the weight of steel* yet they have the same tensile strength as steel piping and can withstand the same degree of abrasion. Being very light, they can be assembled in prefabricated sections which require no special handling equipment. In fact, three men, using Bristol patented couplings, can install or lay 200 ft of 6-inch pressure pipe per hour!

As the pipe bore is glass smooth, flow characteristics are extremely good. And this means that less pumping equipment and smaller-diameter pipes can be used for the same bulk flow.

Bristol Pipe Applications

Bristol pipes have been specially designed for conditions where steel piping corrodes, where installation costs are high, or where prefabricated systems can be used to advantage. Bristol pipes are particularly suitable for chemical plants, oil-well installations and natural-gas pumping stations, and for many other similar applications.

Enquiries should be addressed to:—**Sales Department, Bristol Aeroplane Plastics Limited, Filton, Bristol, England.**

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CPE Diary

JULY 18 TO 22 **Nuclear Technology Survey** at Berkeley, U.S.A. Enrolment limited to persons with **executive or managerial responsibility in industry.** Programme from the Professor of Chemistry, Lawrence radiation laboratory, University of California, Berkeley, Calif., U.S.A.

JULY 18 TO 26 **Tercentenary celebrations of the Royal Society.** Ceremony in the Royal Albert Hall on July 19. Other activities include lectures and a conversazione at Burlington House. Information from Mr. M. Whitchurch, press officer, tercentenary celebrations, I.C.I. Ltd., Millbank, London, S.W.1.

JULY 18 TO 29 Summer programme on the **dynamic behaviour of processes and equipment,** especially where feedback control is utilised, at the Massachusetts Institute of Technology. Details from the Department of Chemical Engineering, M.I.T., Cambridge 39, Mass., U.S.A.

JULY 19 Annual general meeting of the **Plastics Institute** at 2.15 p.m. at the British Plastics Federation, 47-48 Piccadilly, London, W.1. Details from the Plastics Institute, 6 Mandeville Place, London, W.1.

JULY 25 Lecture by **Prof. Linus Pauling** on the **structure of metals and intermetallic compounds** at the Royal Institution, Albemarle Street, London, W.1, at 6.30 p.m. Organised by the metal physics committee of the Institute of Metals. Visitors welcome, no tickets required.

JULY 25 TO AUGUST 6 General Assembly of the International Union of Geodesey and Geophysics including symposium on **atmospheric chemistry and radioactivity** and symposium on **dynamical processes in the atmosphere,** to be held in Helsinki, Finland. Information from the organising committee for the assembly, c/o Institut Geodesique, Bulevardi 40, Helsinki, Finland.

JULY TO AUGUST Symposium on **technical and industrial applications of nuclear energy, including reactor technology and the economics of nuclear energy.** To take place in Brazil. Organised by the Inter-American Nuclear Energy Commission. Details from the executive secretary of the commission, c/o P.A.U., Washington 6, D.C., U.S.A.

AUGUST 1 TO 12 International summer **course in plasma physics** in Denmark. Information from the International Summer Course in Plasma Physics, Danish A.E.C., Research Establishment, Risö, Roskilde, Denmark.

AUGUST 31 TO SEPTEMBER 7 Annual meeting of the **Association for the Advancement of Science** to be held in Cardiff. Details from the society, 18 Adam Street, Adelphi, London, W.C.1.

AUGUST 31 TO SEPTEMBER 11 St. Erik's **Trade Fair** in Stockholm. British participation in fair organised by Trade Fairs and Promotions Ltd., Drury House, Russell Street, Dury Lane, London, W.C.2.

SEPTEMBER 6 TO 17 Conference on the **uses of radioisotopes in the physical sciences and industry** in Copenhagen, Denmark. Sponsored by the International Atomic Energy Agency and U.N.E.S.C.O. Information from I.A.E.A., 11 Kartner Ring, Vienna 1.

SEPTEMBER 11 TO 25 International **trade fair** at Brno, Czechoslovakia. Details from Brno Trade Fair, Vaclavske namnesti, Prague, Czechoslovakia.

SEPTEMBER 13 TO 16 **Instruments and Measurements Conference** in Stockholm. Details from Mr. H. von Koch, Royal Institute of Technology, Stockholm 70, Sweden.

SEPTEMBER 20 TO 23 **Course for directors and senior executives in industry** organised by the U.K.A.E.A. at Wantage. Information from U.K.A.E.A. Isotope School, Wantage Radiation Laboratories, Wantage, Berks.

SEPTEMBER 21 TO OCTOBER 1 **International Factory Equipment Exhibition** at Belle View, Manchester. Information from the organisers, Industrial & Trade Fairs Ltd., Drury House, Russell Street, Drury Lane, London, W.C.2.

SEPTEMBER 22 AND 23 Symposium on **the manufacture and use of some catalysts in the petroleum and petrochemical industries** at the Donnon Laboratories, Grove Street, Liverpool. Organised by the Liverpool section and Chemical Engineering Group of the Society of Chemical Industry. Details from Mr. J. H. Jarwood, Peter Spence & Sons Ltd., Widnes, Lancs.

SEPTEMBER 22 AND 23 Symposium on **the protection of gas plant and equipment from corrosion** at the College of Advanced Technology, Birmingham. Organised jointly by the college, the Institution of Gas Engineers and the Society of Chemical Industry, Corrosion Group. Details from department of chemistry of the college, Suffolk Street, Birmingham 1.

SEPTEMBER 22 TO 24 Conference on **the theory and practice of ultrasonic inspection** to be held at the Queens Hotel, Cheltenham. Organised jointly by the Institute of Physics' Non-Destructive Testing Group, the Society of Non-Destructive Examination and the Non-Destructive Testing Society of Great Britain. Details from the conference secretary, I. M. Barnes, Materials Laboratory, de Havilland Propellers Ltd., Hatfield, Herts.

SEPTEMBER 26 TO 28 Summer school on **the protection of metals by paints** to be held at Battersea College of Technology. Details from the summer school secretary, Battersea College of Technology, London, S.W.11.

SEPTEMBER 29 Robert Horne Memorial Lecture to be delivered by Prof. F. D. Richardson in Bristol. Title: **The Extractions of Metals and the Chemistry of Melts.** Organised by the Society of Chemical Industry, 14 Belgrave Square, London, S.W.1. Details from the secretary.

NOVEMBER 29 TO DECEMBER 2 **The Corrosion and Metal Finishing Exhibition** to be held at Olympia. Organised by Leonard Hill Ltd. Details from the Director, the Corrosion and Metal Finishing Exhibition, 9 Eden Street, London, N.W.1.

International NU-SWIFT News

No. 3

HAVE YOU MET THESE PEOPLE?

39. The UNITED KINGDOM ATOMIC ENERGY AUTHORITY has protected DOUNREAY EXPERIMENTAL REACTOR ESTABLISHMENT in remote CAITHNESS in the North of Scotland with reliable Nu-Swift. The U.K.A.E.A. has some fire risks: The fire in 1957 at their Windscale No. 1 pile cost £1,967,826.

40. KUNGL. SJÖFARTSSTYRELSEN, progressive Sweden's Ministry of Transport, responsible for safety of life at sea according to the 1948 London Convention, was first maritime authority to approve Nu-Swift 30-lb. Dry Powder Extinguisher, Model 1630, for use afloat. Approval is conditional upon spare charges also being carried.

41. Sir HUGH CASSON, famous British architect and exponent of MODERN DESIGN, is one of the many professional men whose premises are protected by Nu-Swift.

42. Rendezvous of the cosmopolitan smart set, the famous Palace Hotel, ST. MORITZ, Switzerland, near the Cresta Run, is now protected by Nu-Swift. So are many of the vessels of the rich GREEK SHIPOWNERS who make the Palace Hotel their winter headquarters.

43. Norwegian WHALE CATCHING SHIPS each year set out from SANDEFJORD, Norway, destined for hazardous adventures in ANTARCTICA. Six or nine months later they return after incredible hardship, but often with the pockets of the crew lined with gold. Most whalers and floating whale factories are equipped with reliable Nu-Swift.

44. In lovely GREENLAND, no longer merely the home of eskimos, and a colony, but part of the kingdom of Denmark, with output from cryolite mines becoming of increasing economic importance, trading posts of the ROYAL GREENLAND TRADING CORPORATION have been equipped with Nu-Swift.

HAVE YOU BEEN TO THESE PLACES?

45. The HOME OFFICE FORENSIC LABORATORY at PRESTON, LANCS., ENGLAND, famous for its fact-searching work in many murder trials, has been equipped with Nu-Swift.

46. Now also available with instructions in ARABIC, Nu-Swift extinguishers can be specified with instructions in this and any of 13 OTHER LANGUAGES at no extra charge.

47. PUNCH, the famous weekly, in recent years brightened by a face-lift, is now also, to prevent possible interruption in publication, protected by Nu-Swift. So are the giant presses in the three cities of publication of the DAILY EXPRESS, printed simultaneously every day in 4,000,000 copies, in LONDON, MANCHESTER and GLASGOW.

48. In GUATEMALA, prosperous Central American State, all the BANKS, except one, are protected by reliable Nu-Swift.

49. SOCIETE NATIONALE DES CHEMINS DE FER BELGES (The BELGIAN STATE RAILWAYS) have, after extensive laboratory tests in BRUSSELS by SOFINA, placed their first substantial orders for Nu-Swift extinguishers.

50. In the unhappy event of another global war, many CIVIL DEFENCE EXPERTS believe that the self-contained Nu-Swift Universal (Royal Navy) 2-gallon Water/CO_2 extinguishers will be of great value in dealing with the many small fires likely to flare up at some distance from a THERMO-NUCLEAR EXPLOSION, at a time when public water supplies will probably be interrupted.

51. Where only a century ago, native warriors, with unexampled ferocity, fought the white man in ZULULAND, local farmers are now forming themselves into Nu-Swift fire-fighting groups of five to pool their Nu-Swift fire posts and jointly combat attacks of the equally ferocious fire fiend. Twenty-nine such groups have so far been organised; they have proved themselves of great value in defeating dangerous SUGAR CANE FIRES.

52. Through the failure of old soda acid extinguishers, CLOVER MEATS Ltd., WATERFORD, Republic of IRELAND, a firm which does a large export business, lost a contract worth £80,000. In consequence, 69 soda acid extinguishers were scrapped and, instead, reliable Nu-Swift was installed to the tune of £1,500.

53. The private railway coach of President KEKKONEN, the first citizen of democratic FINLAND, is now protected by effective and reliable Nu-Swift Dry Powder Extinguisher, Model 1604.

54. 'Guinness is good for you' says the famous slogan of the GUINNESS BREWERY, in Dublin, a national institution in the Republic of IRELAND. The brewery is now protected by Nu-Swift and that, of course, is good for GUINNESS!

55. Founded in 1843, the NATIONAL METSOVIAN POLYTECHNIC SCHOOL in ATHENS, GREECE, the largest and most important scientific institution in South Eastern Europe and the Middle East, has, after exhaustive investigation, decided to standardise on Nu-Swift extinguishers. Initial order was obtained in the teeth of keen local, German, American and Italian competition.

56. To ensure rapid, reliable and efficient operation at sub-zero temperatures, THE PORT OF HELSINKI AUTHORITY, FINLAND, has installed Nu-Swift Dry Powder Extinguishers for the protection of all its buildings and cranes.

DISASTROUS FIRES CRIPPLE INDUSTRY,

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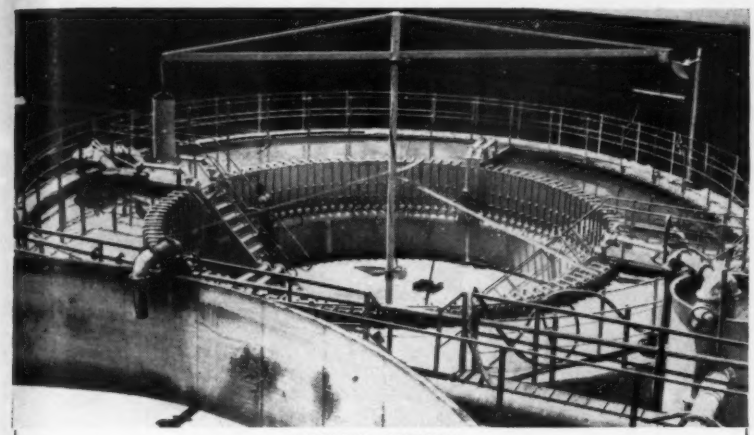
World News



SOUTH AFRICA

Filter plant of the clarifying installation at Western Reefs Gold Mine used for extraction of uranium ore from residual slimes. The circular pipe is 1½-in. ABS Durapipe *made by Durapipe & Fittings Ltd., with elbows, sockets, unions and tees all solvent-welded.*

£ s d

CHEMICAL PLANT COSTS

Cost indices for the month of May 1960 are as follows:

Plant Construction Index: 180.6

Equipment Cost Index: 170.4

(June 1949 = 100)

£ s d

DENMARK

Refinery

A refinery, based on a crude unit with a capacity of 20,000 bbl./day, with vessels designed for a future throughput of 40,000 bbl./day, is to be built at Kalundborg for Dansk Veedol A/S. The complete 'Grass Roots' refinery is to be built by Foster Wheeler Ltd. To minimise costs and reduce the danger of pollution and its effects on local amenities, the refinery will use air fin coolers in place of the more conventional water cooling equipment.

PORTUGAL

Urea plant

An agreement has been signed between Montecatini of U.S.A. and Uniao Fabril do Azoto of Portugal for the construction of a 40,000-metric-tons-p.a. urea plant at Labraidio, near the town of Barreiro on the Tagus River.

The plant is being engineered by the M. W. Kellogg Co. and will incorporate the *Fauser - Montecatini* process—total recycle in the liquid phase which allows the full utilisation of the ammonia feed in the formation of urea. The alternative of partial recycling of ammonia will also be possible. This partial utilisation of the feed for the urea synthesis will permit the production of nitrogen compounds other than urea to accommodate existing market demand for such products.

GREECE

Sulphuric acid plant

Chemical Construction (G.B.) Ltd. are to furnish the design, engineering, equipment and supervision of construction and initial operation of a 376-metric-ton/day sulphur-burning sulphuric acid plant, to be erected at Ptolemais, in the northern part of Greece, for the Greek Ministries of Industry and Coordination, as part of a large nitrogenous fertiliser factory.

UNITED STATES

New merger

Penn-Olin Chemical Co. is now being formed as a joint subsidiary of Pennsalt Chemicals Corporation and Olin Mathieson Chemical Corporation, it was announced recently.

The subsidiary, a $6-million venture, has been organised to produce sodium chlorate and other chlorate compounds, and will be owned equally by the two corporations. Preliminary engineering has been completed, and construction is expected to start soon on a 25,000-ton-p.a. plant at Calvert City.

Pennsalt has produced sodium and potassium chlorate for many years at Portland. Recently it also built an ammonium perchlorate plant in the same area. Olin Mathieson has initiated the development of a process for the generation of chlorine dioxide from sodium chlorate.

This is used in the production of ammonium perchlorate, a solid rocket fuel oxidiser, and in the manufacture of defoliants and weedkillers.

AUSTRALIA

Refinery

B.P. Australia Ltd. will spend between £A8 million to £A10 million building the refinery at Kwinana. It will produce lubricating oils, and will work in conjunction with another refinery already operated by the company at Kwinana.

The new refinery will produce about 100,000 tons of lubricants p.a. for the Australian and export markets. It is already in an advanced state of planning and would bring the company's capital investment in Western Australia to about £A50 million.

Chemical Engineering Fundamentals

(*Concluded from page* 310)

where C_p = the specific heat in cal./g.°C., ρ is the density of g./sq.cm., M = molecular weight and k will be in cal./sec.cm.°C.—due to Weber (see also Reid and Sherwood).

Generally k diminishes with temperature for liquids, thus

$$k = k_{To} \times [1 + a\,(T - To)]$$

where a averages about –0.0012.

The viscosity of liquids decreases rapidly with increasing absolute temperature, according to the law

$$\mu = Ae^{H/T}$$

which is in contrast to the increase of viscosity of a gas ($\mu_{gas} \propto \sqrt{T}$). The exponential term is associated with need to activate a liquid molecule, energetically, before it can move from one position to another.

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Orders and Contracts

Compound mill

A compound mill is being built for the British Extracting Co. Ltd., oil seed crushers and manufacturers of cattle, sheep, pig and poultry foods for Lever's Feeds Ltd. The mill is being built at Bromborough by Holland & Hannen and Cubitts (North West) Ltd. The cost of building and civil engineering work is about £1 million.

Work which is scheduled to be completed by September 1961 will comprise a raw materials warehouse, silo building, production building and finished products warehouse, with administrative buildings and ancillary work.

Water treatment

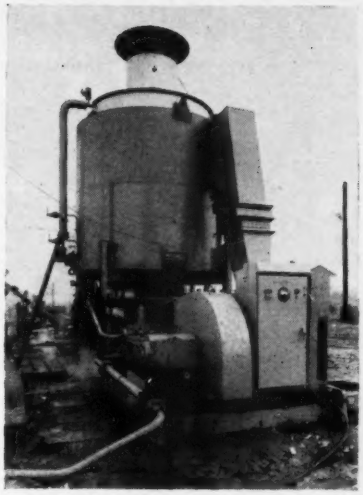
William Boby & Co. Ltd. have been awarded a contract valued at more than £20,000 by Courtaulds for the extension of their *Courtelle* plant at Grimsby. Other orders received by the company totalling over £7,000 are a base exchanger plant for the new boiler house of the South Durham Iron & Steel Co.'s North works, a second demineralisation plant for the research laboratory of Fisons Ltd. and a lime-soda plant for the National Coal Board at Linby Colliery.

Large packaged boilers

Mitchell Engineering Ltd. have received orders for two large packaged boilers, the first for installation at the U.K.A.E.A., Windscale, plant, and the second for service in Venezuela with Shell Petroleum.

The Windscale boiler has been designed for emergency applications, and consequently is able to cut in at very short notice when other steam supplies are inadequate for peak load processes or when they are reduced due to unforeseen circumstances. The boiler, which is of 66,000-lb./hr. evaporation and is automatically controlled, is fired by four *Hamworthy* pressure jet burners, which enable maintenance of individual burner tips without reduction of load when operating. The boiler superheater is of the self-draining *Melesco* type.

The Shell boiler, which has recently been installed, has a special-purpose package vertical coil unit designed to produce steam quickly for supplying saturated steam at 1,000 p.s.i. gauge to Venezuelan oil wells so as to augment the oil recovery.



The packaged boiler recently supplied to Shell, Venezuela, designed to supply saturated steam at 1,000 p.s.i.

The boiler, which is of 22,400-lb./hr. evaporation, is mounted on a heavy skid framed base to facilitate transportation, and is complete with pump, forced draught fan, instrumentation and automatic controls.

The feed supply is very coarse Maracaibo water, which after elementary feed treatment is fed to the boiler unit, necessitating a 15% blowdown.

Vacuum melting furnace

The General Electric Co. Ltd., in association with Vacuum Industrial Applications Ltd. and the British Geco Engineering Co., has secured an order, nearly £100,000 in value, from Jessop-Saville Ltd. for the supply of an induction melting furnace of 1 ton capacity. This is believed to be the largest furnace of its type being manufactured in this country and is scheduled for installation in the company's Brightside works, Sheffield, by the end of the year.

The furnace, which will operate at 1,700°C. is designed for semi-continuous operation and has provision for casting under vacuum. Bulk charging and alloy addition can also be effected without breaking the vacuum.

The power is to be supplied to the furnace at two frequencies, one of which will be used for heating, and the other for stirring the molten metal.

Corrosion and Metal Finishing Exhibition

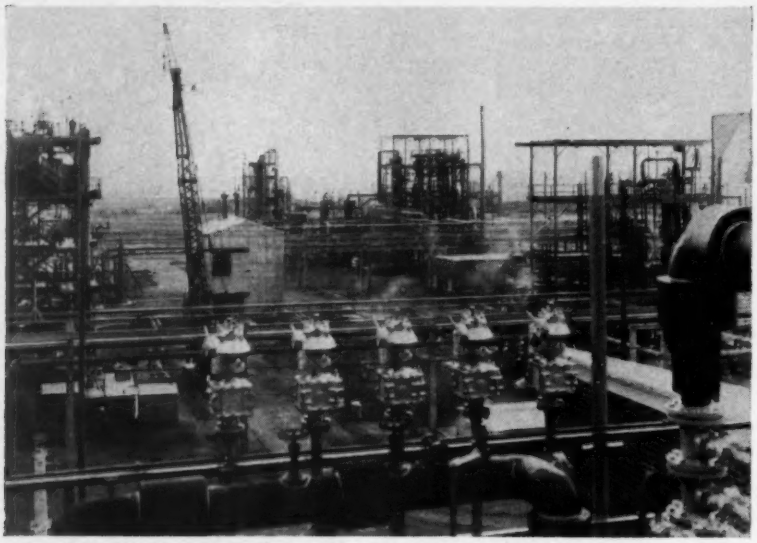
The Organisers of the Corrosion Exhibition have for some time given consideration to a new name that would better express the purpose and content of this important and increasingly popular show. They have taken into account the fact that a great many exhibitors sell products that both protect *and finish* metals. In many cases corrosion-protection and metal finishing are inseparable and implicit. After a great deal of discussion and consultation they have decided to make explicit that which is already implicit and have chosen the amplified name of: The Corrosion and Metal Finishing Exhibition. Thus the Corrosion Exhibition, like all vigorously growing enterprises, enters a new phase of its existence. Everything that made a success of the Corrosion Exhibition is retained. Everything is being done to widen its appeal and draw the right kind of visitors to Olympia from November 29 to December 2.

An encouraging and significant feature is the growing official support for the Exhibition. The Corrosion Group of the S.C.I. have accepted the Organiser's invitation to hold a meeting at Olympia during the Exhibition. Plans are being made for the participation of other professional and technical bodies and Government departments concerned with corrosion and metal finishing. Already the D.S.I.R. and the Atomic Energy Authority are exhibiting.

Publicity for the Exhibition is well under way. It will embrace the total resources of the Leonard Hill technical publications, and the technical and national press, direct mail and editorial features.

Already the Exhibition is heavily booked and with its widened appeal consideration is being given to providing further space in the Empire Hall, Olympia. Intending exhibitors are urged to write or phone now to book their stands. Contact: The Director, The Corrosion and Metal Finishing Exhibition, 9 Eden Street, London, N.W.1, Euston 5911.

Company News



Part of the newly completed Maydown works of the Du Pont Co. (U.K.) Ltd. for the manufacture of neoprene. The picture shows the monomer area photographed from the top of the polymer building.

Anhydrous hydrazine plant

A multi-million dollar plant for the production of anhydrous hydrazine is to be built at Saltville, U.S.A., for the U.S. Air Force by Olin Mathieson Chemical Corporation, by whom the plant will be operated. The corporation is America's only commercial producer of anhydrous hydrazine.

Phthalate ester plant extension

Durham Chemicals Ltd. has extended its manufacturing facilities at Birtley, by the erection and commissioning of a phthalate ester plant for the manufacture of plasticisers for PVC products.

The plant follows the commissioning of plant for liquid stabilisers and paint additives as part of an overall programme to provide supplies of processing chemicals for the paint and thermoplastics industries, both of which are of growing importance in the U.K. economy.

Ammonia and methanol

I.C.I. Ltd. is to make large additions to its plants for manufacturing ammonia and methanol and for making products from ammonia.

A new ammonia plant, with a capacity of 100,000 tons p.a. and associated plants to make urea and fertilisers will be built at I.C.I.'s Severnside works in South Gloucestershire, at a cost of more than £10 millions. These plants are expected to start production during 1963.

The company has also decided to extend the methanol plant at its Heysham works in Lancashire. A new plant to make 30,000 tons p.a. of methanol began production there only a few months ago. A further extension to make an additional 45,000 tons p.a. is now to be built, and is expected to be completed during 1962.

Anglo-German enterprise

Vickers Ltd. and Herr Hans J. Zimmer of Frankfurt am Main recently formed a joint enterprise (Hans J. Zimmer Ltd.), for industrial plant construction, in which each party holds an equal share of the equity.

The company will be active in the entire field of plant design, engineering and construction for the chemical and man-made fibre industries.

The two firms will also hold equal shares in High Polymer & Petrochemical Engineering Ltd.

Neoprene manufacture

First commercial production of neoprene synthetic rubber in the U.K. has started at Maydown works of Du Pont Co. (U.K.) Ltd. near Londonderry.

The works are designed to produce the various types of neoprene rubber and latex in great demand for a variety of end-products.

There are four major production areas, including facilities for the manufacture of nitrogen, hydrogen, hydrogen chloride and other chemicals necessary for the polymerisation process.

Industrial Publications

Vacuum coating. A catalogue devoted entirely to plant suitable for such technical applications of vacuum coating as lens blooming, front surface mirrors, interference filters, cathode ray tubes, quartz crystals, selenium rectifiers and roll coating of tissue for paper capacitors is issued by Edwards High Vacuum Ltd.

Laminates. A film was recently shown in London illustrating the many uses of industrial laminates manufactured by Tufnol Ltd.

PVA emulsions. A booklet which describes the range of *Epok* PVA emulsions has been published by British Resin Products Ltd.

The booklet is divided into three sections: A description of each emulsion in the range, the properties of the emulsions and the applications of emulsions.

The range consists of 15 standard products, nine of which are already plasticised and designed to meet many different requirements.

Sulphuric acid. A booklet has been compiled to correlate information on sulphuric acid manufactured by African Explosives & Chemical Industries Ltd. Its contents include description of grades, properties and uses of sulphuric acid, as well as mentioning appropriate materials of construction, pumps and valves. It should be of interest to both users and manufacturers.

Hydrotropes are compounds which when dissolved in water will assimilate molecules of other compounds normally insoluble in water. The resultant solutions are clear and mobile. A brochure describing properties of *Eltesol* hydrotropes, both in liquid and flake form and mentioning their many applications is available from Marchon Products Ltd.

Vacuum plant. The protection and insulation of electrical and electronic components by encapsulation with resins has enabled designers of equipment to achieve freedom from troubles of many kinds. A new leaflet by Pipework & Engineering (Bristol) Ltd. describes plant required for this operation, which is designed to work at a vacuum as low as 1 mm.